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L8: Entry 1 of 7

File: USPT

Sep 4, 2001

DOCUMENT-IDENTIFIER: US 6284259 B1<sup>1</sup>TITLE: Antimicrobial wipes which provide improved residual benefit versus Gram positive bacteriaAbstract Paragraph Left (1):

The present invention relates to an antimicrobial wipe comprising a porous or absorbent sheet impregnated with an antimicrobial cleansing composition, wherein the antimicrobial cleansing composition comprises from about 0.001% to about 5.0%, by weight of the antimicrobial cleansing composition, of an antimicrobial active; from about 0.05% to about 10%, by weight of the antimicrobial cleansing composition, of an anionic surfactant; from about 0.1% to about 10%, by weight of the antimicrobial cleansing composition, of a proton donating agent; and from about 3% to about 99.85%, by weight of the antimicrobial cleansing composition, water; wherein the composition is adjusted to a pH of from about 3.0 to about 6.0; wherein the antimicrobial cleansing composition has a Gram Positive Residual Effectiveness Index of greater than about 0.5; and wherein the antimicrobial cleansing composition has a Mildness Index of less than about 0.3. The invention also encompasses methods for cleansing skin and providing residual effectiveness versus Gram positive bacteria using these products.

Brief Summary Paragraph Right (7):

Some of these traditional products, especially the hard surface cleaners and surgical disinfectants, utilize high levels of alcohol and/or harsh surfactants which have been shown to dry out and irritate skin tissues. Ideal personal cleansers should gently cleanse the skin, cause little or no irritation, and not leave the skin overly dry after frequent use and preferably should provide a moisturizing benefit to the skin.

Brief Summary Paragraph Right (41):

Anionic surfactants for use in the cleansing compositions include alkyl and alkyl ether sulfates. These materials have the respective formulae  $R_{sup.1}O-SO_{sub.3}M$  and  $R_{sup.1}(CH_{sub.2}H_{sub.4}O)_{sub.x}O-SO_{sub.3}M$ , wherein  $R_{sup.1}$  is a saturated or unsaturated, branched or unbranched alkyl group from about 8 to about 24 carbon atoms,  $x$  is 1 to 10, and  $M$  is a water-soluble cation such as ammonium, sodium, potassium, magnesium, triethanolamine, diethanolamine and monoethanolamine. The alkyl sulfates are typically made by the sulfation of monohydric alcohols (having from about 8 to about 24 carbon atoms) using sulfur trioxide or other known sulfation technique. The alkyl ether sulfates are typically made as condensation products of ethylene oxide and monohydric alcohols (having from about 8 to about 24 carbon atoms) and then sulfated. These alcohols can be derived from fats, e.g., coconut oil or tallow, or can be synthetic. Specific examples of alkyl sulfates which may be used in the cleanser compositions are sodium, ammonium, potassium, magnesium, or TEA salts of lauryl or myristyl sulfate. Examples of alkyl ether sulfates which may be used include ammonium, sodium, magnesium, or TEA laureth-3 sulfate.

Brief Summary Paragraph Right (49):

Still other suitable anionic surfactants are the alkylglyceryl ether sulfonates of the form  $R_{sup.1}-OCH_{sub.2}-C(OH)H-CH_{sub.2}-SO_{sub.3}M$ , wherein  $R_{sup.1}$  is a saturated or unsaturated, branched or unbranched alkyl group from about 8 to about 24 carbon atoms, and  $M$  is a water-soluble cation such as ammonium, sodium, potassium, magnesium, triethanolamine, diethanolamine and monoethanolamine. These can be formed by the reaction of epichlorohydrin and sodium bisulfite with fatty alcohols (having from about 8 to about 24 carbon atoms) or other known methods. One example is sodium cocoglyceryl ether sulfonate.

Brief Summary Paragraph Right (51):

Other anionic materials include phosphates such as monoalkyl, dialkyl, and trialkylphosphate salts formed by the reaction of phosphorous pentoxide with monohydric branched or unbranched alcohols having from about 8 to about 24 carbon atoms. These could also be formed by other known phosphorylation methods. An example from this class of surfactants is sodium mono or dilaurylphosphate.

**Brief Summary Paragraph Right (81):**

In order to achieve the mildness required of the present invention, optional ingredients to enhance the mildness to the skin can be added. These ingredients include cationic and nonionic polymers, co-surfactants, moisturizers and mixtures thereof. Polymers useful herein include polyethylene glycols, polypropylene glycols, hydrolyzed silk proteins, hydrolyzed milk proteins, hydrolyzed keratin proteins, guar hydroxypropyltrimonium chloride, polyquats, silicone polymers and mixtures thereof. When used, the mildness enhancing polymers comprise from about 0.1% to about 1%, preferably from about 0.2% to about 1.0%, and more preferably from about 0.2% to about 0.6%, by weight of the antimicrobial cleansing composition, of the composition. Co-surfactants useful herein include nonionic surfactants such as the Genapol.RTM. 24 series of ethoxylated alcohols, POE(20) sorbitan monooleate (Tween.RTM. 80), polyethylene glycol cocoate and Pluronic.RTM. propylene oxide/ethylene oxide block polymers, and amphoteric surfactants such as alkyl betaines, alkyl sultaines, alkyl amphotacetates, alkyl amphodiacetates, alkyl amphopropionates, and alkyl amphodipropionates. When used, the mildness enhancing cosurfactants comprise from about 20% to about 70%, preferably from about 20% to about 50%, by weight of the anionic surfactant, of the cleansing composition.

**Brief Summary Paragraph Right (82):**

Another group of mildness enhancers are lipid skin moisturizing agents which provide a moisturizing benefit to the user of the cleansing wipe when the lipophilic skin moisturizing agent is deposited to the user's skin. When used in the antimicrobial personal cleansing compositions herein, lipophilic skin moisturizing agents are used, they are employed at a level of about 0.1% to about 30%, preferably from about 0.2% to about 10%, most preferably from about 0.5% to about 5% by weight of the composition.

**Brief Summary Paragraph Right (83):**

In some cases, the lipophilic skin moisturizing agent can desirably be defined in terms of its solubility parameter, as defined by Vaughan in Cosmetics and Toiletries, Vol. 103, p. 47-69, October 1988. A lipophilic skin moisturizing agent having a Vaughan solubility Parameter (VSP) from 5 to 10, preferably from 5.5 to 9 is suitable for use in the antimicrobial cleansing compositions herein.

**Brief Summary Paragraph Right (84):**

A wide variety of lipid type materials and mixtures of materials are suitable for use in the antimicrobial cleansing compositions of the present invention. Preferably, the lipophilic skin conditioning agent is selected from the group consisting of hydrocarbon oils and waxes, silicones, fatty acid derivatives, cholesterol, cholesterol derivatives, di- and tri-glycerides, vegetable oils, vegetable oil derivatives, liquid nondigestible oils such as those described in U.S. Pat. No. 3,600,186 to Mattson; Issued Aug. 17, 1971 and U.S. Pat. No. 4,005,195 and 4,005,196 to Jandacek et al; both issued Jan. 25, 1977, all of which are herein incorporated by reference, or blends of liquid digestible or nondigestible oils with solid polyol polyesters such as those described in U.S. Pat. No. 4,797,300 to Jandacek; issued Jan. 10, 1989; U.S. Pat. Nos. 5,306,514, 5,306,516 and 5,306,515 to Letton; all issued Apr. 26, 1994, all of which are herein incorporated by reference, and acetoglyceride esters, alkyl esters, alkenyl esters, lanolin and its derivatives, milk tri-glycerides, wax esters, beeswax derivatives, sterols, phospholipids and mixtures thereof. Fatty acids, fatty acid soaps and water soluble polyols are specifically excluded from our definition of a lipophilic skin moisturizing agent.

**Brief Summary Paragraph Right (86):**

Silicone Oils: Some examples are dimethicone copolyol, dimethylpolysiloxane, diethylpolysiloxane, high molecular weight dimethicone, mixed C1-C30 alkyl polysiloxane, phenyl dimethicone, dimethiconol, and mixtures thereof. More preferred are non-volatile silicones selected from dimethicone, dimethiconol, mixed C1-C30 alkyl polysiloxane, and mixtures thereof. Nonlimiting examples of silicones useful herein are described in U.S. Pat. No. 5,011,681, to Ciotti et al., issued Apr. 30, 1991, which is incorporated by reference.

**Brief Summary Paragraph Right (89):**

Lanolin and its derivatives are preferred and some examples are lanolin, lanolin oil, lanolin wax, lanolin alcohols, lanolin fatty acids, isopropyl lanolate, acetylated lanolin, acetylated lanolin alcohols, lanolin alcohol linoleate, lanolin alcohol riconoleate.

**Brief Summary Paragraph Right (90):**

It is most preferred when at least 75% of the lipophilic skin conditioning agent is comprised of lipids selected from the group consisting: petrolatum, blends of petrolatum and high molecular weight polybutene, mineral oil, liquid nondigestible oils (e.g. liquid cottonseed sucrose octaesters) or blends of liquid digestible or nondigestible oils with solid polyol polyesters (e.g. sucrose octaesters prepared from C22 fatty acids) wherein the ratio of liquid

digestible or nondigestible oil to solid polyol polyester ranges from about 96:4 to about 80:20, hydrogenated or nonhydrogenated polybutene, microcrystalline wax, polyalkene, paraffin, cerasin, ozokerite, polyethylene, perhydrosqualene; dimethicones, alkyl siloxane, polymethylsiloxane, methylphenylpolysiloxane and mixtures thereof. When as blend of petrolatum and other lipids is used, the ratio of petrolatum to the other selected lipids (hydrogenated or unhydrogenated polybutene or polydecene or mineral oil) is preferably from about 10:1 to about 1:2, more preferably from about 5:1 to about 1:1.

**Brief Summary Paragraph Right (91):**

When a lipophilic skin moisturizing agent is employed as the mildness enhancer in the antimicrobial compositions herein, a stabilizer may also be included at a level ranging from about 0.1% to about 10%, preferably from about 0.1% to about 8%, more preferably from about 0.1% to about 5% by weight of the antimicrobial cleansing composition.

**Brief Summary Paragraph Right (92):**

The stabilizer is used to form a crystalline stabilizing network in the liquid cleansing composition that prevents the lipophilic skin moisturizer agent droplets from coalescing and phase splitting in the product. The network exhibits time dependent recovery of viscosity after shearing (e.g., thixotropy).

**Brief Summary Paragraph Right (97):**

Fumed silica, which is also known as arced silica, is produced by the vapor phase hydrolysis of silicon tetrachloride in a hydrogen oxygen flame. It is believed that the combustion process creates silicone dioxide molecules which condense to form particles. The particles collide, attach and sinter together. The result of this process is a three dimensional branched chain aggregate. Once the aggregate cools below the fusion point of silica, which is about 1710.degree. C., further collisions result in mechanical entanglement of the chains to form agglomerates. Precipitated silicas and silica gels are generally made in aqueous solution. See, Cabot Technical Data Pamphlet TD-100 entitled "CAB-O-SIL.RTM. Untreated Fumed Silica Properties and Functions", October 1993; and Cabot Technical Data Pamphlet TD-104 entitled "CAB-O-SIL.RTM. Fumed Silica in Cosmetic and Personal Care Products", March 1992, both of which are herein incorporated by reference.

**Brief Summary Paragraph Right (102):**

Other known stabilizers, such as fatty acids and fatty alcohols, can also be employed in the compositions herein. Palmitic acid and lauric acid are especially preferred for use herein.

**Brief Summary Paragraph Right (103):**

The compositions of the present invention can comprise a wide range of optional ingredients. The CTFA International Cosmetic Ingredient Dictionary, Sixth Edition, 1995, which is incorporated by reference herein in its entirety, describes a wide variety of nonlimiting cosmetic and pharmaceutical ingredients commonly used in the skin care industry, which are suitable for use in the compositions of the present invention. Nonlimiting examples of functional classes of ingredients are described at page 537 of this reference. Examples of these functional classes include: abrasives, anti-acne agents, anticaking agents, antioxidants, binders, biological additives, bulking agents, chelating agents, chemical additives, colorants, cosmetic astringents, cosmetic biocides, denaturants, drug astringents, emulsifiers, external analgesics, film formers, fragrance components, humectants, opacifying agents, plasticizers, preservatives, propellants, reducing agents, skin bleaching agents, skin-conditioning agents (emollient, humectants, miscellaneous, and occlusive), skin protectants, solvents, foam boosters, hydrotropes, solubilizing agents, suspending agents (nonsurfactant), sunscreen agents, ultraviolet light absorbers, and viscosity increasing agents (aqueous and nonaqueous). Examples of other functional classes of materials useful herein that are well known to one of ordinary skill in the art include solubilizing agents, sequestrants, and keratolytics, and the like.

**Brief Summary Paragraph Right (107):**

Any method suitable for the application of aqueous or aqueous/alcoholic impregnates, including flood coating, spray coating or metered dosing, can be used to impregnate the fibrous webs herein with the antimicrobial cleansing compositions described herein. More specialized techniques, such as Meyer Rod, floating knife or doctor blade, which are typically used to impregnate liquids into absorbent sheets may also be used.

**Brief Summary Paragraph Right (134):**

The Carried CSL 100 Controlled Stress Rheometer is used to determine Shear Index, n, and Consistency, k, of the lipophilic skin moisturizing agent used herein. The determination is performed at 35.degree. C. with the 4 cm 2.degree. cone measuring system typically set with a 51 micron gap and is performed via the programmed application of a shear stress (typically from about 0.06 dynes/sq. cm to about 5,000 dynes/sq. cm) over time. If

this stress results in a deformation of the sample, i.e. strain of the measuring geometry of at least 10<sup>-4</sup> rad/sec, then this rate of strain is reported as a shear rate. These data are used to create a viscosity  $\mu$ . Vs. shear rate  $\gamma$ . flow curve for the material. This flow curve can then be modeled in order to provide a mathematical expression that describes the material's behavior within specific limits of shear stress and shear rate. These results were fitted with the following well accepted power law model (see for instance: Chemical Engineering, by Coulson and Richardson, Pergamon, 1982 or Transport Phenomena by Bird, Stewart and Lightfoot, Wiley, 1960):

Brief Summary Paragraph Left (41):

Consistency (k) and Shear Index (n) of the Lipophilic Skin Moisturizing Agent

Brief Summary Paragraph Type 0 (9):

Benzyl Alcohol

Brief Summary Paragraph Type 0 (18):

Phenethyl Alcohol

Brief Summary Paragraph Type 0 (24):

Dichlorobenzyl Alcohol

Other Reference Publication (8):

Blank, Irvin H., PhD, Measurement of pH of the Skin Surface, (1939), The Journal of Investigative Dermatology, vol. 2, pp. 75-79.

CLAIMS:

1. An antimicrobial wipe comprising a porous or absorbent sheet impregnated with an antimicrobial cleansing composition, wherein the antimicrobial cleansing composition comprises:

- a. from about 0.001% to about 5.0%, by weight of the antimicrobial cleansing composition, of antimicrobial active;
- b. from about 0.05% to about 10%, by weight of the antimicrobial cleansing composition, of anionic surfactant;
- c. from about 0.1% to about 10%, by weight of the antimicrobial cleansing composition, of proton donating agent; and
- d. from about 3% to about 99.85%, by weight of the antimicrobial cleansing composition, water;

wherein the antimicrobial cleansing composition is adjusted to a pH of from about 3.0 to about 6.0; and wherein the antimicrobial cleansing composition has a Gram Positive Residual Effectiveness Index of greater than about 0.5.

2. An antimicrobial wipe comprising according to claim 1 wherein the antimicrobial cleansing composition has a Mildness Index of greater than about 0.3.

3. An antimicrobial wipe according to claim 2 wherein the antimicrobial active is selected from the group consisting of triclosan, triclocarban, piroctone olamine, PCMX, ZPT, natural essential oils and their key chemical components and mixtures thereof.

4. An antimicrobial wipe according to claim 3 wherein the antimicrobial active is triclosan.

5. An antimicrobial wipe according to claim 3 wherein the anionic surfactant has a Microtox Response Index of less than about 150.

6. An antimicrobial wipe according to claim 3 wherein the proton donating agent is an organic acid having a Buffering Capacity of greater than about 0.01.

7. An antimicrobial wipe according to claim 5 wherein the proton donating agent is an organic acid having a Buffering Capacity of greater than about 0.01.

8. An antimicrobial wipe according to claim 3 wherein the proton donating agent is a mineral acid.
9. An antimicrobial wipe according to claim 3 wherein the composition is adjusted to a pH of from about 3.5 to about 5.0.
10. An antimicrobial wipe according to claim 7 wherein the antimicrobial cleansing composition is adjusted to a pH of from about 3.5 to about 5.0.
11. An antimicrobial wipe according to claim 9 wherein the ratio of the amount of non-anionic surfactants to the amount of anionic surfactant in the antimicrobial cleansing composition is less than 1:1.
12. An antimicrobial wipe according to claim 10 wherein the ratio of the amount of non-anionic surfactants to the amount of anionic surfactant in the antimicrobial cleansing composition is less than 1:1.
13. An antimicrobial wipe comprising a porous or absorbent sheet impregnated with an antimicrobial cleansing composition, wherein the antimicrobial cleansing composition comprises:
- a. from about 0.001% to about 5.0%, by weight of the antimicrobial cleansing composition, of antimicrobial active;
  - b. from about 0.05% to about 10%, by weight of the antimicrobial cleansing composition, of anionic surfactant;
  - c. from about 0.1% to about 10%, by weight of the antimicrobial cleansing composition, of protein donating agent;
  - d. from about 0.1% to about 30% by weight of the antimicrobial cleansing composition of lipophilic skin moisturizing agent; and
  - e. from about 3% to about 99.85%, by weight of the antimicrobial cleansing composition, water;
- wherein the antimicrobial cleansing composition is adjusted to a pH of from about 3.0 to about 6.0; wherein the antimicrobial cleansing composition has a Gram Positive Residual Effectiveness Index of greater than about 1.0; and wherein the antimicrobial cleansing composition has a Mildness Index of greater than about 0.4.
14. An antimicrobial wipe according to claim 13 wherein the antimicrobial cleansing composition comprises from about 0.1% to about 2%, by weight of the antimicrobial cleansing composition, of the anionic surfactant.
15. An antimicrobial wipe according to claim 14 wherein the proton donating agent is an organic acid and has a buffering capacity of greater than 0.01.
16. An antimicrobial wipe according to claim 15 wherein the anionic surfactant is selected from the group consisting of sodium and ammonium alkyl sulfates and ether sulfates having chain lengths of predominantly 12 and 14 carbon atoms, olefin sulfates having chain lengths of predominantly 14 and 16 carbon atoms, and paraffin sulfonates having an average chain length of from 13 to 17 carbon atoms, and mixtures thereof.
17. An antimicrobial wipe according to claim 15 wherein the antimicrobial cleansing composition is adjusted to a pH of from about 3.5 to about 5.0.
18. An antimicrobial wipe according to claim 17 wherein the proton donating agent is selected from the group comprising adipic acid, tartaric acid, citric acid, maleic acid, malic acid, succinic acid, glycolic acid, glutaric acid, benzoic acid, malonic acid, salicylic acid, gluconic acid, polymeric acids, their salts, and mixtures thereof.
19. A method for providing residual effectiveness against Gram positive bacteria comprising rubbing the antimicrobial wipe of claim 1 on human skin, wherein the antimicrobial cleansing composition is applied to the skin in a safe and effective amount.
20. A method for providing residual effectiveness against Gram positive bacteria comprising rubbing the antimicrobial wipe of claim 13 on human skin, wherein the antimicrobial cleansing composition is applied to the skin in a safe and effective amount.

21. A method for treating acne comprising rubbing the antimicrobial wipe of claim 1 on human skin, wherein the antimicrobial cleansing composition is applied to the skin in a safe and effective amount.

22. An antimicrobial wipe according to claim 17 wherein the proton donating agent is selected from the group consisting of straight-chain poly(acrylic) acids and copolymers thereof, cross-linked poly(acrylic) acids having a molecular weight of less than about 250,000, poly (.alpha.-hydroxy) acids and copolymers thereof, poly(methacrylic) acid and copolymers thereof, polysulfonic acid and copolymers thereof, carrageen acid, carboxy methyl cellulose, and alginic acid.

23. An antimicrobial wipe according to claim 1 wherein the antimicrobial cleansing composition comprises from about 0.05% to about 2%, by weight of the antimicrobial cleansing composition, of the anionic surfactant.

24. An antimicrobial wipe according to claim 1 wherein the antimicrobial cleansing composition comprises from about 0.1% to about 1%, by weight of the antimicrobial cleansing composition, of the anionic surfactant.

25. An antimicrobial wipe according to claim 1 wherein the ratio of the amount of non-anionic surfactants to the amount of anionic surfactant in the antimicrobial cleansing composition is less than 1:2.

26. An antimicrobial wipe according to claim 1 wherein the ratio of the amount of non-anionic surfactants to the amount of anionic surfactant in the antimicrobial cleansing composition is less than 1:4.

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File: USPT

Jul 10, 2001

DOCUMENT-IDENTIFIER: US 6258368 B1

TITLE: Antimicrobial wipesAbstract Paragraph Left (1):

The present invention relates to an antimicrobial wipe comprising a porous or absorbent sheet impregnated with an antimicrobial cleansing composition, wherein the antimicrobial cleansing composition comprises from about 0.001% to about 5.0%, by weight of the antimicrobial cleansing composition, of an antimicrobial active; from about 0.05% to about 10%, by weight of the antimicrobial cleansing composition, of an anionic surfactant; from about 0.1% to about 10%, by weight of the antimicrobial cleansing composition, of a proton donating agent; and from about 3% to about 99.85%, by weight of the antimicrobial cleansing composition, water; wherein the composition is adjusted to a pH of from about 3.0 to about 6.0. The invention also encompasses methods for cleansing skin, reducing the number of germs on the skin, and providing residual effectiveness versus Gram positive and Gram negative bacteria using these products.

Brief Summary Paragraph Right (7):

Some of these traditional products, especially the hard surface cleaners and surgical disinfectants, utilize high levels of alcohol and/or harsh surfactants which have been shown to dry out and irritate skin tissues. Ideal personal cleansers should gently cleanse the skin, cause little or no irritation, and not leave the skin overly dry after frequent use and preferably should provide a moisturizing benefit to the skin.

Brief Summary Paragraph Right (40):

Anionic surfactants for use in the cleansing compositions include alkyl and alkyl ether sulfates. These materials have the respective formulae  $R_{sup.1}O-SO_{sub.3}M$  and  $R_{sup.1}(CH_{sub.2}H_{sub.4}O)_{sub.x}O-SO_{sub.3}M$ , wherein  $R_{sup.1}$  is a saturated or unsaturated, branched or unbranched alkyl group from about 8 to about 24 carbon atoms,  $x$  is 1 to 10, and  $M$  is a water-soluble cation such as ammonium, sodium, potassium, magnesium, triethanolamine, diethanolamine and monoethanolamine. The alkyl sulfates are typically made by the sulfation of monohydric alcohols (having from about 8 to about 24 carbon atoms) using sulfur trioxide or other known sulfation technique. The alkyl ether sulfates are typically made as condensation products of ethylene oxide and monohydric alcohols (having from about 8 to about 24 carbon atoms) and then sulfated. These alcohols can be derived from fats, e.g., coconut oil or tallow, or can be synthetic. Specific examples of alkyl sulfates which may be used in the cleanser compositions are sodium, ammonium, potassium, magnesium, or TEA salts of lauryl or myristyl sulfate. Examples of alkyl ether sulfates which may be used include ammonium, sodium, magnesium, or TEA laureth-3 sulfate.

Brief Summary Paragraph Right (48):

Still other suitable anionic surfactants are the alkylglyceryl ether sulfonates of the form  $R_{sup.1}OCH_{sub.2}C(OH)H-CH_{sub.2}SO_{sub.3}M$ , wherein  $R_{sup.1}$  is a saturated or unsaturated, branched or unbranched alkyl group from about 8 to about 24 carbon atoms, and  $M$  is a water-soluble cation such as ammonium, sodium, potassium, magnesium, triethanolamine, diethanolamine and monoethanolamine. These can be formed by the reaction of epichlorohydrin and sodium bisulfite with fatty alcohols (having from about 8 to about 24 carbon atoms) or other known methods. One example is sodium cocoglyceryl ether sulfonate.

Brief Summary Paragraph Right (50):

Other anionic materials include phosphates such as monoalkyl, dialkyl, and trialkylphosphate salts formed by the reaction of phosphorous pentoxide with monohydric branched or unbranched alcohols having from about 8 to about 24 carbon atoms. These could also be formed by other known phosphorylation methods. An example from this class of surfactants is sodium mono or dilaurylphosphate.

**Brief Summary Paragraph Right (70):**

In order to achieve the mildness required of the present invention, optional ingredients to enhance the mildness to the skin can be added. These ingredients include cationic and nonionic polymers, co-surfactants, moisturizers and mixtures thereof. Polymers useful herein include polyethylene glycols, polypropylene glycols, hydrolyzed silk proteins, hydrolyzed milk proteins, hydrolyzed keratin proteins, guar hydroxypropyltrimonium chloride, polyquats, silicone polymers and mixtures thereof. When used, the mildness enhancing polymers comprise from about 0.1% to about 1%, preferably from about 0.2% to about 1.0%, and more preferably from about 0.2% to about 0.6%, by weight of the antimicrobial cleansing composition, of the composition. Co-surfactants useful herein include nonionic surfactants such as the Genapol.RTM. 24 series of ethoxylated alcohols, POE(20) sorbitan monooleate (Tween.RTM. 80), polyethylene glycol cocoate and Pluronic.RTM. propylene oxide/ethylene oxide block polymers, and amphoteric surfactants such as alkyl betaines, alkyl sultaines, alkyl amphotacetates, alkyl amphodiacetates, alkyl amphopropionates, and alkyl amphodipropionates. When used, the mildness enhancing cosurfactants comprise from about 20% to about 70%, preferably from about 20% to about 50%, by weight of the anionic surfactant, of the cleansing composition.

**Brief Summary Paragraph Right (71):**

Another group of mildness enhancers are lipid skin moisturizing agents which provide a moisturizing benefit to the user of the cleansing wipe when the lipophilic skin moisturizing agent is deposited to the user's skin. When used in the antimicrobial compositions herein, lipophilic skin moisturizing agents are used, they are employed at a level of about 0.1% to about 30%, preferably from about 0.2% to about 10%, most preferably from about 0.5% to about 5% by weight of the composition.

**Brief Summary Paragraph Right (72):**

In some cases, the lipophilic skin moisturizing agent can desirably be defined in terms of its solubility parameter, as defined by Vaughan in Cosmetics and Toiletries, Vol. 103, p. 47-69, October 1988. A lipophilic skin moisturizing agent having a Vaughan solubility Parameter (VSP) from 5 to 10, preferably from 5.5 to 9 is suitable for use in the antimicrobial cleansing compositions herein.

**Brief Summary Paragraph Right (73):**

A wide variety of lipid type materials and mixtures of materials are suitable for use in the antimicrobial cleansing compositions of the present invention. Preferably, the lipophilic skin conditioning agent is selected from the group consisting of hydrocarbon oils and waxes, silicones, fatty acid derivatives, cholesterol, cholesterol derivatives, di- and tri-glycerides, vegetable oils, vegetable oil derivatives, liquid nondigestible oils such as those described in U.S. Pat. No. 3,600,186 to Mattson; Issued Aug. 17, 1971 and U.S. Pat. Nos. 4,005,195 and 4,005,196 to Jandacek et al; both issued Jan. 25, 1977, all of which are herein incorporated by reference, or blends of liquid digestible or nondigestible oils with solid polyol polyesters such as those described in U.S. Pat. No. 4,797,300 to Jandacek; issued Jan. 10, 1989; U.S. Pat. Nos. 5,306,514, 5,306,516 and 5,306,515 to Letton; all issued Apr. 26, 1994, all of which are herein incorporated by reference, and acetoglyceride esters, alkyl esters, alkenyl esters, lanolin and its derivatives, milk tri-glycerides, wax esters, beeswax derivatives, sterols, phospholipids and mixtures thereof. Fatty acids, fatty acid soaps and water soluble polyols are specifically excluded from our definition of a lipophilic skin moisturizing agent.

**Brief Summary Paragraph Right (75):**

Silicone Oils: Some examples are dimethicone copolyol, dimethylpolysiloxane, diethylpolysiloxane, high molecular weight dimethicone, mixed C1-C30 alkyl polysiloxane, phenyl dimethicone, dimethiconol, and mixtures thereof. More preferred are non-volatile silicones selected from dimethicone, dimethiconol, mixed C1-C30 alkyl polysiloxane, and mixtures thereof. Nonlimiting examples of silicones useful herein are described in U.S. Pat. No. 5,011,681, to Ciotti et al., issued Apr. 30, 1991, which is incorporated by reference.

**Brief Summary Paragraph Right (78):**

Lanolin and its derivatives are preferred and some examples are lanolin, lanolin oil, lanolin wax, lanolin alcohols, lanolin fatty acids, isopropyl lanolate, acetylated lanolin, acetylated lanolin alcohols, lanolin alcohol linoleate, lanolin alcohol riconoleate.

**Brief Summary Paragraph Right (79):**

It is most preferred when at least 75% of the lipophilic skin conditioning agent is comprised of lipids selected from the group consisting: petrolatum, blends of petrolatum and high molecular weight polybutene, mineral oil, liquid nondigestible oils (e.g. liquid cottonseed sucrose octaesters) or blends of liquid digestible or nondigestible oils with solid polyol polyesters (e.g. sucrose octaesters prepared from C22 fatty acids) wherein the ratio of liquid digestible or nondigestible oil to solid polyol polyester ranges from about 96:4 to about 80:20, hydrogenated or



nonhydrogenated polybutene, microcrystalline wax, polyalkene, paraffin, cerasin, ozokerite, polyethylene, perhydrosqualene; dimethicones, alkyl siloxane, polymethylsiloxane, methylphenylpolysiloxane and mixtures thereof. When as blend of petrolatum and other lipids is used, the ratio of petrolatum to the other selected lipids (hydrogenated or unhydrogenated polybutene or polydecene or mineral oil) is preferably from about 10:1 to about 1:2, more preferably from about 5:1 to about 1:1.

Brief Summary Paragraph Right (80):

When a lipophilic skin moisturizing agent is employed as the mildness enhancer in the antimicrobial compositions herein, a stabilizer may also be included at a level ranging from about 0.1% to about 10%, preferably from about 0.1% to about 8%, more preferably from about 0.1% to about 5% by weight of the antimicrobial cleansing composition.

Brief Summary Paragraph Right (81):

The stabilizer is used to form a crystalline stabilizing network in the liquid cleansing composition that prevents the lipophilic skin moisturizer agent droplets from coalescing and phase splitting in the product. The network exhibits time dependent recovery of viscosity after shearing (e.g., thixotropy).

Brief Summary Paragraph Right (86):

Fumed silica, which is also known as arced silica, is produced by the vapor phase hydrolysis of silicon tetrachloride in a hydrogen oxygen flame. It is believed that the combustion process creates silicone dioxide molecules which condense to form particles. The particles collide, attach and sinter together. The result of this process is a three dimensional branched chain aggregate. Once the aggregate cools below the fusion point of silica, which is about 1710.degree. C., further collisions result in mechanical entanglement of the chains to form agglomerates. Precipitated silicas and silica gels are generally made in aqueous solution. See, Cabot Technical Data Pamphlet TD-100 entitled "CAB-O-SIL.RTM. Untreated Fumed Silica Properties and Functions", October 1993, and Cabot Technical Data Pamphlet TD-104 entitled "CAB-O-SIL.RTM. Fumed Silica in Cosmetic and Personal Care Products", March 1992, both of which are herein incorporated by reference.

Brief Summary Paragraph Right (91):

Other known stabilizers, such as fatty acids and fatty alcohols, can also be employed in the compositions herein. Palmitic acid and lauric acid are especially preferred for use herein.

Brief Summary Paragraph Right (92):

The compositions of the present invention can comprise a wide range of optional ingredients. The CTFA International Cosmetic Ingredient Dictionary, Sixth Edition, 1995, which is incorporated by reference herein in its entirety, describes a wide variety of nonlimiting cosmetic and pharmaceutical ingredients commonly used in the skin care industry, which are suitable for use in the compositions of the present invention. Nonlimiting examples of functional classes of ingredients are described at page 537 of this reference. Examples of these functional classes include: abrasives, anti-acne agents, anticaking agents, antioxidants, binders, biological additives, bulking agents, chelating agents, chemical additives, colorants, cosmetic astringents, cosmetic biocides, denaturants, drug astringents, emulsifiers, external analgesics, film formers, fragrance components, humectants, opacifying agents, plasticizers, preservatives, propellants, reducing agents, skin bleaching agents, skin-conditioning agents (emollient, humectants, miscellaneous, and occlusive), skin protectants, solvents, foam boosters, hydrotropes, solubilizing agents, suspending agents (nonsurfactant), sunscreen agents, ultraviolet light absorbers, and viscosity increasing agents (aqueous and nonaqueous). Examples of other functional classes of materials useful herein that are well known to one of ordinary skill in the art include solubilizing agents, sequestrants, and keratolytics, and the like.

Brief Summary Paragraph Right (93):

Any method suitable for the application of aqueous or aqueous/alcoholic impregnates, including flood coating, spray coating or metered dosing, can be used to impregnate the fibrous webs herein with the antimicrobial cleansing compositions described herein. More specialized techniques, such as Meyer Rod, floating knife or doctor blade, which are typically used to impregnate liquids into absorbent sheets may also be used.

Brief Summary Paragraph Type 0 (9):

Benzyl Alcohol

Brief Summary Paragraph Type 0 (18):

Phenethyl Alcohol

**Brief Summary Paragraph Type 0 (24):****Dichlorobenzyl Alcohol****CLAIMS:**

1. An antimicrobial wipe comprising a porous or absorbent sheet impregnated with an antimicrobial cleansing composition, wherein the antimicrobial cleansing composition comprises:

- a. from about 0.001% to about 5.0%, by weight of the antimicrobial cleansing composition, of an antimicrobial active;
- b. from about 0.05% to about 10%, by weight of the antimicrobial cleansing composition, of an anionic surfactant;
- c. from about 0.1% to about 10%, by weight of the antimicrobial cleansing composition, of a proton donating agent; and
- d. from about 3% to about 99.85%, by weight of the antimicrobial cleansing composition, water;

wherein the antimicrobial cleansing composition is adjusted to a pH of from about 3.0 to about 6.0.

2. An antimicrobial wipe according to claim 1 wherein the antimicrobial active is selected from the group consisting of triclosan, triclocarban, piroctone olamine, PCMX, ZPT, natural essential oils and their key ingredients, and mixtures thereof.

3. An antimicrobial wipe according to claim 2 wherein the proton donating agent is an organic acid having a Buffering Capacity of greater than about 0.005.

4. An antimicrobial wipe according to claim 3 wherein the anionic surfactant is selected from the group consisting of sodium and ammonium alkyl sulfates and ether sulfates having chain lengths of predominantly 12 and 14 carbon atoms, olefin sulfates having chain lengths of predominantly 14 and 16 carbon atoms, and paraffin sulfonates having an average chain length of from 13 to 17 carbon atoms, and mixtures thereof.

5. An antimicrobial wipe according to claim 4 wherein the proton donating agent is selected from the group comprising adipic acid, tartaric acid, citric acid, maleic acid, malic acid, succinic acid, glycolic acid, glutaric acid, benzoic acid, malonic acid, salicylic acid, gluconic acid, polyacrylic acid, their salts, and mixtures thereof.

6. An antimicrobial wipe according to claim 5 wherein the ratio of the amount of non-anionic surfactants to the amount of anionic surfactant in the antibacterial cleansing composition is less than 1:1.

7. An antimicrobial wipe according to claim 6 wherein the antimicrobial active is present at a level ranging from about 0.05% to about 2%, the anionic surfactant is present at a level of from about 0.1% to about 4%, and the proton donating agent is present at a level of from about 0.5% to about 8%, by weight of the antimicrobial cleansing composition.

8. An antimicrobial wipe according to claim 7 wherein the antimicrobial cleansing composition is adjusted to a pH of from about 3.5 to about 5.0.

9. An antimicrobial wipe according to claim 8 wherein the antimicrobial active is present at a level ranging from about 0.1% to about 1%, the anionic surfactant is present at a level of from about 0.2% to about 1%, and the proton donating agent is present at a level of from about 1% to about 4%, by weight of the antimicrobial cleansing composition.

10. An antimicrobial wipe according to claim 9 wherein the pH is adjusted to from about 3.5 to about 4.5.

11. An antimicrobial wipe according to claim 10 wherein the antimicrobial active is Triclosan.

12. An antimicrobial wipe according to claim 11 wherein the proton donating agent is selected from the group consisting of malic acid, malonic acid, citric acid, succinic acid and lactic acid.

13. An antimicrobial wipe according to claim 12 wherein the anionic surfactant is ammonium lauryl sulfate.
14. An antimicrobial wipe according to claim 1 wherein the antimicrobial cleansing composition further comprises a mildness enhancing agent.
15. An antimicrobial wipe according to claim 14 wherein the mildness enhancing agent is selected from the group consisting of from about 0.1% to about 1.0%, by weight of the antimicrobial cleansing composition, of a mildness enhancing polymer, from about 20% to about 70%, by weight of the anionic surfactant, of a mildness enhancing co-surfactant, and mixtures thereof.
16. An antimicrobial wipe according to claim 14 wherein the mildness enhancing agent is from about 0.1% to about 30%, by weight of the antimicrobial cleansing composition, of a lipophilic skin moisturizing agent.
17. An antimicrobial wipe according to claim 1 wherein the antimicrobial cleansing composition further comprises from about 0.1% to about 10%, by weight of the antimicrobial cleansing composition, of acidic surfactant.
18. An antimicrobial wipe according to claim 1 wherein the antimicrobial cleansing composition comprises from about 0.15% to about 2%, by weight of the antimicrobial cleansing composition, of salicylic acid proton donating agent.
19. A method for providing residual effectiveness against transient Gram negative bacteria, improved residual effectiveness against Gram positive bacteria and improved immediate reduction of germs on human skin comprising contacting the skin with the antimicrobial wipe of claim 1 to apply a safe and effective amount of the antimicrobial cleansing composition on the skin.
20. A method for providing residual effectiveness against transient Gram negative bacteria, improved residual effectiveness against Gram positive bacteria and improved immediate reduction of germs on human skin comprising contacting the skin with the antimicrobial wipe of claim 7 to apply a safe and effective amount of the antimicrobial cleansing composition on the skin.
21. A method for providing residual effectiveness against transient Gram negative bacteria, improved residual effectiveness against Gram positive bacteria and improved immediate reduction of germs on human skin comprising contacting the skin with the antimicrobial wipe of claim 13 to apply a safe and effective amount of the antimicrobial cleansing composition on the skin.
22. A method for providing residual effectiveness against transient Gram negative bacteria, improved residual effectiveness against Gram positive bacteria and improved immediate reduction of germs on human skin comprising contacting the skin with the antimicrobial wipe of claim 16 to apply a safe and effective amount of the antimicrobial cleansing composition on the skin.
23. A method for providing residual effectiveness against transient Gram negative bacteria, improved residual effectiveness against Gram positive bacteria and improved immediate reduction of germs on human skin comprising contacting the skin with the antimicrobial wipe of claim 17 to apply a safe and effective amount of the antimicrobial cleansing composition on the skin.
24. A method for providing residual effectiveness against transient Gram negative bacteria, improved residual effectiveness against Gram positive bacteria and improved immediate reduction of germs on human skin comprising contacting the skin with the antimicrobial wipe of claim 18 a safe and effective amount of the antimicrobial cleansing composition on the skin.
25. A method for treating acne comprising contacting the skin with the antimicrobial wipe of claim 1 to apply a safe and effective amount of the antimicrobial cleansing composition on the skin.
26. An antimicrobial wipe according to claim 1, wherein the antimicrobial cleansing composition comprises from about 0.1% to about 4%, by weight of the antimicrobial cleansing composition, of the anionic surfactant.
27. An antimicrobial wipe according to claim 1, wherein the antimicrobial cleansing composition comprises from about 0.2% to about 1%, by weight of the antimicrobial cleansing composition, of the anionic surfactant.

28. An antimicrobial wipe according to claim 1, wherein the ratio of the amount of non-anionic surfactant to the amount of anionic surfactant in the antibacterial cleansing composition is less than 1:1.

29. An antimicrobial wipe according to claim 1, wherein the ratio of the amount of non-anionic surfactant to the amount of anionic surfactant in the antibacterial cleansing composition is less than 1:2.

30. An antimicrobial wipe according to claim 1, wherein the ratio of the amount of non-anionic surfactant to the amount of anionic surfactant in the antibacterial cleansing composition is less than 1:4.

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File: USPT

Mar 6, 2001

DOCUMENT-IDENTIFIER: US 6197315 B1

TITLE: Antimicrobial wipes which provide improved residual benefit versus gram negative bacteriaAbstract Paragraph Left (1):

The present invention relates to an antimicrobial wipe comprising a porous or absorbent sheet impregnated with an antimicrobial cleansing composition, wherein the antimicrobial cleansing composition comprises from about 0.001% to about 5.0%, by weight of the antimicrobial cleansing composition, of an antimicrobial active; from about 0.05% to about 10%, by weight of the antimicrobial cleansing composition, of an anionic surfactant; from about 0.1% to about 10%, by weight of the antimicrobial cleansing composition, of a proton donating agent; and from about 3% to about 99.85%, by weight of the antimicrobial cleansing composition, water; wherein the composition is adjusted to a pH of from about 3.0 to about 6.0; wherein the antimicrobial cleansing composition has a Gram Negative Residual Effectiveness Index of greater than about 0.3. The invention also relates to these antibacterial wipes wherein the antimicrobial cleansing composition has a Mildness Index of greater than about 0.3. The invention also encompasses methods for cleansing skin and providing residual effectiveness versus Gram negative bacteria using these products.

Brief Summary Paragraph Right (7):

Some of these traditional products, especially the hard surface cleaners and surgical disinfectants, utilize high levels of alcohol and/or harsh surfactants which have been shown to dry out and irritate skin tissues. Ideal personal cleansers should gently cleanse the skin, cause little or no irritation, and not leave the skin overly dry after frequent use and preferably should provide a moisturizing benefit to the skin.

Brief Summary Paragraph Right (41):

Anionic surfactants for use in the cleansing compositions include alkyl and alkyl ether sulfates. These materials have the respective formulae  $R_{sup.1}O-SO_{sub.3}M$  and  $R_{sup.1}(CH_{sub.2}H_{sub.4}O)_{sub.x}O-SO_{sub.3}M$ , wherein  $R_{sup.1}$  is a saturated or unsaturated, branched or unbranched alkyl group from about 8 to about 24 carbon atoms,  $x$  is 1 to 10, and  $M$  is a water-soluble cation such as ammonium, sodium, potassium, magnesium, triethanolamine, diethanolamine and monoethanolamine. The alkyl sulfates are typically made by the sulfation of monohydric alcohols (having from about 8 to about 24 carbon atoms) using sulfur trioxide or other known sulfation technique. The alkyl ether sulfates are typically made as condensation products of ethylene oxide and monohydric alcohols (having from about 8 to about 24 carbon atoms) and then sulfated. These alcohols can be derived from fats, e.g., coconut oil or tallow, or can be synthetic. Specific examples of alkyl sulfates which may be used in the cleanser compositions are sodium, ammonium, potassium, magnesium, or TEA salts of lauryl or myristyl sulfate. Examples of alkyl ether sulfates which may be used include ammonium, sodium, magnesium, or TEA laureth-3 sulfate.

Brief Summary Paragraph Right (49):

Still other suitable anionic surfactants are the alkylglyceryl ether sulfonates of the form  $R_{sup.1}-OCH_{sub.2}-C(OH)H-CH_{sub.2}-SO_{sub.3}M$ , wherein  $R_{sup.1}$  is a saturated or unsaturated, branched or unbranched alkyl group from about 8 to about 24 carbon atoms, and  $M$  is a water-soluble cation such as ammonium, sodium, potassium, magnesium, triethanolamine, diethanolamine and monoethanolamine. These can be formed by the reaction of epichlorohydrin and sodium bisulfite with fatty alcohols (having from about 8 to about 24 carbon atoms) or other known methods. One example is sodium cocoglyceryl ether sulfonate.

Brief Summary Paragraph Right (51):

Other anionic materials include phosphates such as monoalkyl, dialkyl, and trialkylphosphate salts formed by the reaction of phosphorous pentoxide with monohydric branched or unbranched alcohols having from about 8 to about 24 carbon atoms. These could also be formed by other known phosphorylation methods. An example from

this class of surfactants is sodium mono or dilaurylphosphate.

**Brief Summary Paragraph Right (80):**

In order to achieve the mildness required of the present invention, optional ingredients to enhance the mildness to the skin can be added. These ingredients include cationic and nonionic polymers, co-surfactants, moisturizers and mixtures thereof. Polymers useful herein include polyethylene glycols, polypropylene glycols, hydrolyzed silk proteins, hydrolyzed milk proteins, hydrolyzed keratin proteins, guar hydroxypropyltrimonium chloride, polyquats, silicone polymers and mixtures thereof. When used, the mildness enhancing polymers comprise from about 0.1% to about 1%, preferably from about 0.2% to about 1.0%, and more preferably from about 0.2% to about 0.6%, by weight of the antimicrobial cleansing composition, of the composition. Co-surfactants useful herein include nonionic surfactants such as the Genapol.RTM. 24 series of ethoxylated alcohols, POE(20) sorbitan monooleate (Tween.RTM. 80), polyethylene glycol cocoate and Pluronic.RTM. propylene oxide/ethylene oxide block polymers, and amphoteric surfactants such as alkyl betaines, alkyl sultaines, alkyl amphotacetates, alkyl amphodiacetates, alkyl amphopropionates, and alkyl amphodipropionates. When used, the mildness enhancing cosurfactants comprise from about 20% to about 70%, preferably from about 20% to about 50%, by weight of the anionic surfactant, of the cleansing composition.

**Brief Summary Paragraph Right (81):**

Another group of mildness enhancers are lipid skin moisturizing agents which provide a moisturizing benefit to the user of the cleansing wipe when the lipophilic skin moisturizing agent is deposited to the user's skin. When used in the antimicrobial personal cleansing compositions herein, lipophilic skin moisturizing agents are used, they are employed at a level of about 0.1% to about 30%, preferably from about 0.2% to about 10%, most preferably from about 0.5% to about 5% by weight of the composition.

**Brief Summary Paragraph Right (82):**

In some cases, the lipophilic skin moisturizing agent can desirably be defined in terms of its solubility parameter, as defined by Vaughan in Cosmetics and Toiletries, Vol. 103, p. 47-69, October 1988. A lipophilic skin moisturizing agent having a Vaughan solubility Parameter (VSP) from 5 to 10, preferably from 5.5 to 9 is suitable for use in the antimicrobial cleansing compositions herein.

**Brief Summary Paragraph Right (83):**

A wide variety of lipid type materials and mixtures of materials are suitable for use in the antimicrobial cleansing compositions of the present invention. Preferably, the lipophilic skin conditioning agent is selected from the group consisting of hydrocarbon oils and waxes, silicones, fatty acid derivatives, cholesterol, cholesterol derivatives, di and tri-glycerides, vegetable oils, vegetable oil derivatives, liquid nondigestible oils such as those described in U.S. Pat. No. 3,600,186 to Mattson; Issued Aug. 17, 1971 and U.S. Pat. No. 4,005,195 and U.S. Pat. No. 4,005,196 to Jandacek et al; both issued Jan. 25, 1977, all of which are herein incorporated by reference, or blends of liquid digestible or nondigestible oils with solid polyol polyesters such as those described in U.S. Pat. No. 4,797,300 to Jandacek; issued Jan. 10, 1989; U.S. Pat. Nos. 5,306,514, 5,306,516 and 5,306,515 to Letton; all issued Apr. 26, 1994, all of which are herein incorporated by reference, and acetoglyceride esters, alkyl esters, alkenyl esters, lanolin and its derivatives, milk tri-glycerides, wax esters, beeswax derivatives, sterols, phospholipids and mixtures thereof. Fatty acids, fatty acid soaps and water soluble polyols are specifically excluded from our definition of a lipophilic skin moisturizing agent.

**Brief Summary Paragraph Right (85):**

Silicone Oils: Some examples are dimethicone copolyol, dimethylpolysiloxane, diethylpolysiloxane, high molecular weight dimethicone, mixed C1-C30 alkyl polysiloxane, phenyl dimethicone, dimethiconol, and mixtures thereof. More preferred are non-volatile silicones selected from dimethicone, dimethiconol, mixed C1-C30 alkyl polysiloxane, and mixtures thereof. Nonlimiting examples of silicones useful herein are described in U.S. Pat. No. 5,011,681, to Ciotti et al., issued Apr. 30, 1991, which is incorporated by reference.

**Brief Summary Paragraph Right (88):**

Lanolin and its derivatives are preferred and some examples are lanolin, lanolin oil, lanolin wax, lanolin alcohols, lanolin fatty acids, isopropyl lanolate, acetylated lanolin, acetylated lanolin alcohols, lanolin alcohol linoleate, lanolin alcohol riconoleate.

**Brief Summary Paragraph Right (89):**

It is most preferred when at least 75% of the lipophilic skin conditioning agent is comprised of lipids selected from the group consisting: petrolatum, blends of petrolatum and high molecular weight polybutene, mineral oil, liquid nondigestible oils (e.g. liquid cottonseed sucrose octaesters) or blends of liquid digestible or nondigestible

oils with solid polyol polyesters (e.g. sucrose octaesters prepared from C22 fatty acids) wherein the ratio of liquid digestible or nondigestible oil to solid polyol polyester ranges from about 96:4 to about 80:20, hydrogenated or nonhydrogenated polybutene, microcrystalline wax, polyalkene, paraffin, cerasin, ozokerite, polyethylene, perhydrosqualene; dimethicones, alkyl siloxane, polymethylsiloxane, methylphenylpolysiloxane and mixtures thereof. When as blend of petrolatum and other lipids is used, the ratio of petrolatum to the other selected lipids (hydrogenated or unhydrogenated polybutene or polydecene or mineral oil) is preferably from about 10:1 to about 1:2, more preferably from about 5:1 to about 1:1.

**Brief Summary Paragraph Right (90):**

When a lipophilic skin moisturizing agent is employed as the mildness enhancer in the antimicrobial compositions herein, a stabilizer may also be included at a level ranging from about 0.1% to about 10%, preferably from about 0.1% to about 8%, more preferably from about 0.1% to about 5% by weight of the antimicrobial cleansing composition.

**Brief Summary Paragraph Right (91):**

The stabilizer is used to form a crystalline stabilizing network in the liquid cleansing composition that prevents the lipophilic skin moisturizer agent droplets from coalescing and phase splitting in the product. The network exhibits time dependent recovery of viscosity after shearing (e.g., thixotropy).

**Brief Summary Paragraph Right (96):**

Fumed silica, which is also known as arced silica, is produced by the vapor phase hydrolysis of silicon tetrachloride in a hydrogen oxygen flame. It is believed that the combustion process creates silicone dioxide molecules which condense to form particles. The particles collide, attach and sinter together. The result of this process is a three dimensional branched chain aggregate. Once the aggregate cools below the fusion point of silica, which is about 1710.degree. C., further collisions result in mechanical entanglement of the chains to form agglomerates. Precipitated silicas and silica gels are generally made in aqueous solution. See, Cabot Technical Data Pamphlet TD-100 entitled "CAB-O-SIL.RTM. Untreated Fumed Silica Properties and Functions", October 1993, and Cabot Technical Data Pamphlet TD-104 entitled "CAB-O-SIL.RTM. Fumed Silica in Cosmetic and Personal Care Products", March 1992, both of which are herein incorporated by reference.

**Brief Summary Paragraph Right (101):**

Other known stabilizers, such as fatty acids and fatty alcohols, can also be employed in the compositions herein. Palmitic acid and lauric acid are especially preferred for use herein.

**Brief Summary Paragraph Right (102):**

The compositions of the present invention can comprise a wide range of optional ingredients. The CTFA International Cosmetic Ingredient Dictionary, Sixth Edition, 1995, which is incorporated by reference herein in its entirety, describes a wide variety of nonlimiting cosmetic and pharmaceutical ingredients commonly used in the skin care industry, which are suitable for use in the compositions of the present invention. Nonlimiting examples of functional classes of ingredients are described at page 537 of this reference. Examples of these functional classes include: abrasives, anti-acne agents, anticaking agents, antioxidants, binders, biological additives, bulking agents, chelating agents, chemical additives, colorants, cosmetic astringents, cosmetic biocides, denaturants, drug astringents, emulsifiers, external analgesics, film formers, fragrance components, humectants, opacifying agents, plasticizers, preservatives, propellants, reducing agents, skin bleaching agents, skin-conditioning agents (emollient, humectants, miscellaneous, and occlusive), skin protectants, solvents, foam boosters, hydrotropes, solubilizing agents, suspending agents (nonsurfactant), sunscreen agents, ultraviolet light absorbers, and viscosity increasing agents (aqueous and nonaqueous). Examples of other functional classes of materials useful herein that are well known to one of ordinary skill in the art include solubilizing agents, sequestrants, and keratolytics, and the like.

**Brief Summary Paragraph Right (106):**

Any method suitable for the application of aqueous or aqueous/alcoholic impregnates, including flood coating, spray coating or metered dosing, can be used to impregnate the fibrous webs herein with the antimicrobial cleansing compositions described herein. More specialized techniques, such as Meyer Rod, floating knife or doctor blade, which are typically used to impregnate liquids into absorbent sheets may also be used.

**Brief Summary Paragraph Right (128):**

The Carrimed CSL 100 Controlled Stress Rheometer is used to determine Shear Index, n, and Consistency, k, of the lipophilic skin moisturizing agent used herein. The determination is performed at 35.degree. C. with the 4 cm 2.degree. cone measuring system typically set with a 51 micron gap and is performed via the programmed

application of a shear stress (typically from about 0.06 dynes/sq. cm to about 5,000 dynes/sq. cm) over time. If this stress results in a deformation of the sample, i.e. strain of the measuring geometry of at least 10<sup>-4</sup> rad/sec, then this rate of strain is reported as a shear rate. These data are used to create a viscosity .mu. Vs. shear rate .gamma.' flow curve for the material. This flow curve can then be modeled in order to provide a mathematical expression that describes the material's behavior within specific limits of shear stress and shear rate. These results were fitted with the following well accepted power law model (see for instance: Chemical Engineering, by Coulson and Richardson, Pergamon, 1982 or Transport Phenomena by Bird, Stewart and Lightfoot, Wiley, 1960):

Brief Summary Paragraph Left (25):

CONSISTENCY (k) AND SHEAR INDEX (n) OF THE LIPOPHILIC SKIN MOISTURIZING AGENT

Brief Summary Paragraph Type 0 (9):

Benzyl Alcohol

Brief Summary Paragraph Type 0 (18):

Phenethyl Alcohol

Brief Summary Paragraph Type 0 (24):

Dichlorobenzyl Alcohol

Other Reference Publication (9):

Blank, Irvin H., PhD, Measurement of pH of the Skin Surface, (1939), The Journal of Investigative Dermatology, vol. 2, pp. 75-79.

Other Reference Publication (22):

Kabara, Jon J., Structure-function relationships of surfactants as antimicrobial agents, (1978), Journal of the Society of Cosmetic Chemists, 29, pp. 733-741.

CLAIMS:

1. An antimicrobial wipe comprising a porous or absorbent sheet impregnated with an antimicrobial cleansing composition, wherein the antimicrobial cleansing composition comprises the following components:

- a. from about 0.001% to about 5.0%, by weight of the antimicrobial cleansing composition, of an antimicrobial active;
- b. from about 0.05% to about 10%, by weight of the antimicrobial cleansing composition, of an anionic surfactant;
- c. from about 0.1% to about 10%, by weight of the antimicrobial cleansing composition, of a proton donating agent; and
- d. from about 3% to about 99.85%, by weight of the antimicrobial cleansing composition, water;

wherein the composition is adjusted to a pH of from about 3.0 to about 6.0; and

wherein the antimicrobial cleansing composition has a Gram Negative Residual Effectiveness Index of greater than about 0.3.

2. An antimicrobial wipe according to claim 1, wherein the antimicrobial cleansing composition has a Mildness Index of greater than about 0.3.

3. An antimicrobial wipe according to claim 2 wherein the antimicrobial active is selected from the group consisting of triclosan, triclocarbon, pivoctone alamine, PCMX, ZPT, natural essential oils and their key ingredients, and mixtures thereof.

4. An antimicrobial wipe according to claim 3 wherein the antimicrobial active is triclosan.

5. An antimicrobial wipe according to claim 3 wherein the anionic surfactant has a Microtox Response Index of



less than about 150.

6. An antimicrobial wipe according to claim 3 wherein the proton donating agent is an organic acid having a Buffering Capacity of greater than about 0.005.

7. An antimicrobial wipe according to claim 5 wherein the proton donating agent is an organic acid having a Buffering Capacity of greater than about 0.005.

8. An antimicrobial wipe according to claim 3 wherein the proton donating agent is a mineral acid.

9. An antimicrobial wipe according to claim 3 wherein the composition is adjusted to a pH of from about 3.5 to about 5.0.

10. An antimicrobial wipe according to claim 7 wherein the composition is adjusted to a pH of from about 3.5 to about 5.0.

11. An antimicrobial wipe according to claim 9 wherein the ratio of the amount of non-anionic surfactants to the amount of anionic surfactant in the antimicrobial cleansing composition is less than 1:1.

12. An antimicrobial wipe according to claim 10 wherein the ratio of the amount of non-anionic surfactants to the amount of anionic surfactant in the antimicrobial cleansing composition is less than 1:1.

13. An antimicrobial wipe comprising a porous or absorbent sheet impregnated with an antimicrobial cleansing composition, wherein the antimicrobial cleansing composition comprises the following individual components:

a. from about 0.001% to about 5.0%, by weight of the antimicrobial cleansing composition, of an antimicrobial active;

b. from about 0.05% to about 10%, by weight of the antimicrobial cleansing composition, of an anionic surfactant;

c. from about 0.1% to about 10%, by weight of the antimicrobial cleansing composition, of a proton donating agent;

d. from about 0.1% to about 30% by weight of the antimicrobial cleansing composition of a lipophilic skin moisturizing agent; and

e. from about 3% to about 98%, by weight of the antimicrobial cleansing composition, water;

wherein the composition is adjusted to a pH of from about 3.0 to about 6.0; wherein the antimicrobial cleansing composition has a Gram Negative Residual Effectiveness Index of greater than about 1.0; and wherein the antimicrobial cleansing composition has a Mildness Index of greater than about 0.4.

14. An antimicrobial wipe according to claim 13 comprising from about 0.1% to about 2%, by weight of the antimicrobial cleansing composition, of the anionic surfactant.

15. An antimicrobial wipe according to claim 14 wherein the proton donating agent is an organic acid and has a buffering capacity of greater than 0.01.

16. An antimicrobial wipe according to claim 15 wherein the anionic surfactant is selected from the group consisting of sodium and ammonium alkyl sulfates and ether sulfates having chain lengths of predominantly 12 and 14 carbon atoms, olefin sulfates having chain lengths of predominantly 14 and 16 carbon atoms, and paraffin sulfonates having an average chain length of from 13 to 17 carbon atoms, and mixtures thereof.

17. An antimicrobial wipe according to claim 16 wherein the composition is adjusted to a pH of from about 3.5 to about 5.0.

18. An antimicrobial wipe according to claim 17 wherein the proton donating agent is selected from the group comprising adipic acid, tartaric acid, citric acid, maleic acid, malic acid, succinic acid, glycolic acid, glutaric acid, benzoic acid, malonic acid, salicylic acid, gluconic acid, polyacrylic acid, their salts, and mixtures thereof.

19. A method for providing improved residual effectiveness against Gram negative bacteria comprising rubbing the antimicrobial wipe of claim 1 on human skin, wherein the antimicrobial cleansing composition is applied to the skin in a safe and effective amount.

20. A method for providing residual effectiveness against Gram negative bacteria comprising rubbing the antimicrobial wipe of claim 13 on human skin, wherein the antimicrobial cleansing composition is applied to the skin in a safe and effective amount.

21. A method for treating acne comprising rubbing the antimicrobial wipe of claim 1 on human skin, wherein the antimicrobial composition is applied to the skin in a safe and effective amount.

22. An antimicrobial wipe according to claim 1 wherein the antimicrobial cleansing composition comprises from about 0.05% to about 5%, of the anionic surfactant.

23. An antimicrobial wipe according to claim 1 wherein the antimicrobial cleansing composition comprises from about 0.05% to about 2%, of the anionic surfactant.

24. An antimicrobial wipe according to claim 1 wherein the antimicrobial cleansing composition comprises from about 0.2% to about 1%, of the anionic surfactant.

25. An antimicrobial wipe according to claim 1 wherein the weight ratio of the amount of non-anionic surfactants to the amount of anionic surfactant in the antimicrobial cleansing composition is less than about 1:2.

26. An antimicrobial wipe according to claim 1 wherein the weight ratio of the amount of non-anionic surfactants to the amount of anionic surfactant in the antimicrobial cleansing composition is less than about 1:4.

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File: USPT

Feb 6, 2001

DOCUMENT-IDENTIFIER: US.6183763 B1

TITLE: Antimicrobial wipes which provide improved immediate germ reduction**Abstract Paragraph Left (1):**

The present invention relates to an antimicrobial wipe effective against Gram positive bacteria, Gram negative bacteria, fungi, yeasts, molds, and viruses comprising a porous or absorbent sheet impregnated with an antimicrobial cleansing composition, wherein the antimicrobial cleansing composition comprises from about 0.001% to about 5.0%, by weight of the antimicrobial cleansing composition, of an antimicrobial active; from about 0.05% to about 10%, by weight of the antimicrobial cleansing composition, of an anionic surfactant; from about 0.1% to about 10%, by weight of the antimicrobial cleansing composition, of a proton donating agent; and from about 3% to about 99.85%, by weight of the antimicrobial cleansing composition, water; wherein the composition is adjusted to a pH of from about 3.0 to about 6.0; wherein the antimicrobial cleansing composition has an One-wash Immediate Germ Reduction Index of greater than about 1.3; and wherein the rinse-off antimicrobial cleansing composition has a Mildness Index of greater than 0.3. The present invention also relates to methods for removing germs from the skin using the antimicrobial wipes described herein.

**Brief Summary Paragraph Right (7):**

Some of these antimicrobial products, especially the hard surface cleaners and surgical disinfectants, utilize high levels of alcohol and/or harsh surfactants which have been shown to dry out and irritate skin tissues. Dial.RTM. bar soap has been found to provide from 2.5 to 3.0 log reduction in bacteria in one wash, as measured by the HCPHWT. However, Dial.RTM. can be drying to the skin with repeated use. Hibiclens.RTM. Surgical Scrub provides 2.5 to 3.0 log reduction in germs in one wash, however it utilizes a potent cationic antibacterial agent, chlorohexidine, which has product safety concerns. Ideal personal cleansers should gently cleanse the skin, cause little or no irritation, and not leave the skin overly dry after frequent use and preferably should provide a moisturizing benefit to the skin.

**Brief Summary Paragraph Right (40):**

Anionic surfactants for use in the cleansing compositions include alkyl and alkyl ether sulfates. These materials have the respective formulae  $R_{sup.1}O-SO_{sub.3}M$  and  $R_{sup.1}(CH_{sub.2}H_{sub.4}O)_{sub.x}O-SO_{sub.3}M$ , wherein  $R_{sup.1}$  is a saturated or unsaturated, branched or unbranched alkyl group from about 8 to about 24 carbon atoms,  $x$  is 1 to 10, and  $M$  is a water-soluble cation such as ammonium, sodium, potassium, magnesium, triethanolamine, diethanolamine and monoethanolamine. The alkyl sulfates are typically made by the sulfation of monohydric alcohols (having from about 8 to about 24 carbon atoms) using sulfur trioxide or other known sulfation technique. The alkyl ether sulfates are typically made as condensation products of ethylene oxide and monohydric alcohols (having from about 8 to about 24 carbon atoms) and then sulfated. These alcohols can be derived from fats, e.g., coconut oil or tallow, or can be synthetic. Specific examples of alkyl sulfates which may be used in the cleanser compositions are sodium, ammonium, potassium, magnesium, or TEA salts of lauryl or myristyl sulfate. Examples of alkyl ether sulfates which may be used include ammonium, sodium, magnesium, or TEA laureth-3 sulfate.

**Brief Summary Paragraph Right (48):**

Still other suitable anionic surfactants are the alkylglyceryl ether sulfonates of the form  $R_{sup.1}-OCH_{sub.2}-C(OH)H-CH_{sub.2}-SO_{sub.3}M$ , wherein  $R_{sup.1}$  is a saturated or unsaturated, branched or unbranched alkyl group from about 8 to about 24 carbon atoms, and  $M$  is a water-soluble cation such as ammonium, sodium, potassium, magnesium, triethanolamine, diethanolamine and monoethanolamine. These can be formed by the reaction of epichlorohydrin and sodium bisulfite with fatty alcohols (having from about 8 to about 24 carbon atoms) or other known methods. One example is sodium cocoglyceryl ether sulfonate.

**Brief Summary Paragraph Right (50):**

Other anionic materials include phosphates such as monoalkyl, dialkyl, and trialkylphosphate salts formed by the reaction of phosphorous pentoxide with monohydric branched or unbranched alcohols having from about 8 to about 24 carbon atoms. These could also be formed by other known phosphorylation methods. An example from this class of surfactants is sodium mono or dilaurylphosphate.

**Brief Summary Paragraph Right (79):**

In order to achieve the mildness required of the present invention, optional ingredients to enhance the mildness to the skin can be added. These ingredients include cationic and nonionic polymers, co-surfactants, moisturizers and mixtures thereof. Polymers useful herein include polyethylene glycols, polypropylene glycols, hydrolyzed silk proteins, hydrolyzed milk proteins, hydrolyzed keratin proteins, guar hydroxypropyltrimonium chloride, polyquats, silicone polymers and mixtures thereof. When used, the mildness enhancing polymers comprise from about 0.1% to about 1%, preferably from about 0.2% to about 1.0%, and more preferably from about 0.2% to about 0.6%, by weight of the antimicrobial cleansing composition, of the composition. Co-surfactants useful herein include nonionic surfactants such as the Genapol.RTM. 24 series of ethoxylated alcohols, POE(20) sorbitan monooleate (Tween.RTM. 80), polyethylene glycol cocoate and Pluronic.RTM. propylene oxide/ethylene oxide block polymers, and amphoteric surfactants such as alkyl betaines, alkyl sultaines, alkyl amphoacetates, alkyl amphodiacetates, alkyl amphopropionates, and alkyl amphodipropionates. When used, the mildness enhancing co-surfactants comprise from about 20% to about 70%, preferably from about 20% to about 50%, by weight of the anionic surfactant, of the cleansing composition.

**Brief Summary Paragraph Right (80):**

Another group of mildness enhancers are lipid skin moisturizing agents which provide a moisturizing benefit to the user of the cleansing wipe when the lipophilic skin moisturizing agent is deposited to the user's skin. When used in the antimicrobial personal cleansing compositions herein, lipophilic skin moisturizing agents are used, they are employed at a level of about 0.1% to about 30%, preferably from about 0.2% to about 10%, most preferably from about 0.5% to about 5% by weight of the composition.

**Brief Summary Paragraph Right (81):**

In some cases, the lipophilic skin moisturizing agent can desirably be defined in terms of its solubility parameter, as defined by Vaughan in Cosmetics and Toiletries, Vol. 103, p. 47-69, October 1988. A lipophilic skin moisturizing agent having a Vaughan solubility Parameter (VSP) from 5 to 10, preferably from 5.5 to 9 is suitable for use in the antimicrobial cleansing compositions herein.

**Brief Summary Paragraph Right (82):**

A wide variety of lipid type materials and mixtures of materials are suitable for use in the antimicrobial cleansing compositions of the present invention. Preferably, the lipophilic skin conditioning agent is selected from the group consisting of hydrocarbon oils and waxes, silicones, fatty acid derivatives, cholesterol, cholesterol derivatives, di- and tri-glycerides, vegetable oils, vegetable oil derivatives, liquid nondigestible oils such as those described in U.S. Pat. No. 3,600,186 to Mattson; Issued Aug. 17, 1971 and U.S. Pat. Nos. 4,005,195 and 4,005,196 to Jandacek et al; both issued Jan. 25, 1977, all of which are herein incorporated by reference, or blends of liquid digestible or nondigestible oils with solid polyol polyesters such as those described in U.S. Pat. No. 4,797,300 to Jandacek; issued Jan. 10, 1989; U.S. Pat. Nos. 5,306,514, 5,306,516 and 5,306,515 to Letton; all issued Apr. 26, 1994, all of which are herein incorporated by reference, and acetoglyceride esters, alkyl esters, alkenyl esters, lanolin and its derivatives, milk tri-glycerides, wax esters, beeswax derivatives, sterols, phospholipids and mixtures thereof. Fatty acids, fatty acid soaps and water soluble polyols are specifically excluded from our definition of a lipophilic skin moisturizing agent.

**Brief Summary Paragraph Right (84):**

Silicone Oils: Some examples are dimethicone copolyol, dimethylpolysiloxane, diethylpolysiloxane, high molecular weight dimethicone, mixed C1-C30 alkyl polysiloxane, phenyl dimethicone, dimethiconol, and mixtures thereof. More preferred are non-volatile silicones selected from dimethicone, dimethiconol, mixed C1-C30 alkyl polysiloxane, and mixtures thereof. Nonlimiting examples of silicones useful herein are described in U.S. Pat. No. 5,011,681, to Ciotti et al., issued Apr. 30, 1991, which is incorporated by reference.

**Brief Summary Paragraph Right (87):**

Lanolin and its derivatives are preferred and some examples are lanolin, lanolin oil, lanolin wax, lanolin alcohols, lanolin fatty acids, isopropyl lanolate, acetylated lanolin, acetylated lanolin alcohols, lanolin alcohol linoleate, lanolin alcohol riconoleate.

**Brief Summary Paragraph Right (88):**

It is most preferred when at least 75% of the lipophilic skin conditioning agent is comprised of lipids selected from the group consisting: petrolatum, blends of petrolatum and high molecular weight polybutene, mineral oil, liquid nondigestible oils (e.g. liquid cottonseed sucrose octaesters) or blends of liquid digestible or nondigestible oils with solid polyol polyesters (e.g. sucrose octaesters prepared from C22 fatty acids) wherein the ratio of liquid digestible or nondigestible oil to solid polyol polyester ranges from about 96:4 to about 80:20, hydrogenated or nonhydrogenated polybutene, microcrystalline wax, polyalkene, paraffin, cerasin, ozokerite, polyethylene, perhydrosqualene; dimeticones, alkyl siloxane, polymethylsiloxane, methylphenylpolysiloxane and mixtures thereof. When as blend of petrolatum and other lipids is used, the ratio of petrolatum to the other selected lipids (hydrogenated or unhydrogenated polybutene or polydecene or mineral oil) is preferably from about 10:1 to about 1:2, more preferably from about 5:1 to about 1:1.

**Brief Summary Paragraph Right (89):**

When a lipophilic skin moisturizing agent is employed as the mildness enhancer in the antimicrobial compositions herein, a stabilizer may also be included at a level ranging from about 0.1% to about 10%, preferably from about 0.1% to about 8%, more preferably from about 0.1% to about 5% by weight of the antimicrobial cleansing composition.

**Brief Summary Paragraph Right (90):**

The stabilizer is used to form a crystalline stabilizing network in the liquid cleansing composition that prevents the lipophilic skin moisturizer agent droplets from coalescing and phase splitting in the product. The network exhibits time dependent recovery of viscosity after shearing (e.g., thixotropy).

**Brief Summary Paragraph Right (95):**

Fumed silica, which is also known as arced silica, is produced by the vapor phase hydrolysis of silicon tetrachloride in a hydrogen oxygen flame. It is believed that the combustion process creates silicone dioxide molecules which condense to form particles. The particles collide, attach and sinter together. The result of this process is a three dimensional branched chain aggregate. Once the aggregate cools below the fusion point of silica, which is about 1710.degree. C., further collisions result in mechanical entanglement of the chains to form agglomerates. Precipitated silicas and silica gels are generally made in aqueous solution. See, Cabot Technical Data Pamphlet TD-100 entitled "CAB-O-SIL.RTM. Untreated Fumed Silica Properties and Functions", October 1993, and Cabot Technical Data Pamphlet TD-104 entitled "CAB-O-SIL.RTM. Fumed Silica in Cosmetic and Personal Care Products", March 1992, both of which are herein incorporated by reference.

**Brief Summary Paragraph Right (100):**

Other known stabilizers, such as fatty acids and fatty alcohols, can also be employed in the compositions herein. Palmitic acid and lauric acid are especially preferred for use herein.

**Brief Summary Paragraph Right (101):**

The compositions of the present invention can comprise a wide range of optional ingredients. The CTFA International Cosmetic Ingredient Dictionary, Sixth Edition, 1995, which is incorporated by reference herein in its entirety, describes a wide variety of nonlimiting cosmetic and pharmaceutical ingredients commonly used in the skin care industry, which are suitable for use in the compositions of the present invention. Nonlimiting examples of functional classes of ingredients are described at page 537 of this reference. Examples of these functional classes include: abrasives, anti-acne agents, anticaking agents, antioxidants, binders, biological additives, bulking agents, chelating agents, chemical additives, colorants, cosmetic astringents, cosmetic biocides, denaturants, drug astringents, emulsifiers, external analgesics, film formers, fragrance components, humectants, opacifying agents, plasticizers, preservatives, propellants, reducing agents, skin bleaching agents, skin-conditioning agents (emollient, humectants, miscellaneous, and occlusive), skin protectants, solvents, foam boosters, hydrotropes, solubilizing agents, suspending agents (nonsurfactant), sunscreen agents, ultraviolet light absorbers, and viscosity increasing agents (aqueous and nonaqueous). Examples of other functional classes of materials useful herein that are well known to one of ordinary skill in the art include solubilizing agents, sequestrants, and keratolytics, and the like.

**Brief Summary Paragraph Right (105):**

Any method suitable for the application of aqueous or aqueous/alcoholic impregnates, including flood coating, spray coating or metered dosing, can be used to impregnate the fibrous webs herein with the antimicrobial cleansing compositions described herein. More specialized techniques, such as Meyer Rod, floating knife or doctor blade, which are typically used to impregnate liquids into absorbent sheets may also be used.

**Brief Summary Paragraph Right (130):**

The Carrimed CSL 100 Controlled Stress Rheometer is used to determine Shear Index, n, and Consistency, k, of the lipophilic skin moisturizing agent used herein. The determination is performed at 35.degree. C. with the 4 cm 2.degree. cone measuring system typically set with a 51 micron gap and is performed via the programmed application of a shear stress (typically from about 0.06 dynes/sq. cm to about 5,000 dynes/sq. cm) over time. If this stress results in a deformation of the sample, i.e. strain of the measuring geometry of at least 10-4 rad/sec, then this rate of strain is reported as a shear rate. These data are used to create a viscosity .mu. Vs. shear rate .gamma.' flow curve for the material. This flow curve can then be modeled in order to provide a mathematical expression that describes the material's behavior within specific limits of shear stress and shear rate. These results were fitted with the following well accepted power law model (see for instance: Chemical Engineering, by Coulson and Richardson, Pergamon, 1982 or Transport Phenomena by Bird, Stewart and Lightfoot, Wiley, 1960):

**Brief Summary Paragraph Left (34):**

CONSISTENCY (k) AND SHEAR INDEX (n) OF THE LIPOPHILIC SKIN MOISTURIZING AGENT

**Brief Summary Paragraph Type 1 (9):**

Benzyl Alcohol

**Brief Summary Paragraph Type 1 (18):**

Phenethyl Alcohol

**Brief Summary Paragraph Type 1 (24):**

Dichlorobenzyl Alcohol

**Other Reference Publication (8):**

Blank, Irvin H., PhD, Measurement of pH of the Skin Surface, (1939), The Journal of Investigative Dermatology, vol. 2, pp. 75-79.

**Other Reference Publication (21):**

Kabara, Jon J., Structure-function relationships of surfactants as antimicrobial agents, (1978), Journal of the Society of Cosmetic Chemists, 29, pp. 733-741.

**CLAIMS:**

1. An antimicrobial wipe effective against Gram positive bacteria, Gram negative bacteria, fungi, yeasts, molds, and viruses comprising a porous or absorbent sheet impregnated with an antimicrobial cleansing composition, wherein the antimicrobial cleansing composition comprises the following individual components:

- a. from about 0.001% to about 5.0%, by weight of the antimicrobial cleansing composition, of an antimicrobial active;
- b. from about 0.05% to about 10%, by weight of the antimicrobial cleansing composition, of an anionic surfactant;
- c. from about 0.1% to about 10%, by weight of the antimicrobial cleansing composition, of a proton donating agent; and
- d. from about 3% to about 99.85%, by weight of the antimicrobial cleansing composition, water;

wherein the composition is adjusted to a pH of from about 3.0 to about 6.0;

wherein the antimicrobial cleansing composition exhibits a One-wash Immediate Germ Reduction Index of greater than about 1.3; and wherein the antimicrobial cleansing composition has a Mildness Index of greater than 0.3.

2. An antimicrobial wipe according to claim 1 wherein the antimicrobial active is selected from the group consisting of triclosan, triclocarban, piroctone olamine, PCMX, ZPT, natural essential oils and their key ingredients, and mixtures thereof.

3. An antimicrobial wipe according to claim 2 wherein the antimicrobial active is triclosan.
4. An antimicrobial wipe according to claim 2 wherein the anionic surfactant has a Microtox Response Index of less than about 150.
5. An antimicrobial wipe according to claim 2 wherein the proton donating agent is an organic acid having a Buffering Capacity of greater than about 0.005.
6. An antimicrobial wipe according to claim 2 wherein the proton donating agent is a mineral acid.
7. An antimicrobial wipe according to claim 5 wherein the composition is adjusted to a pH of from about 3.5 to about 5.0.
8. An antimicrobial wipe according to claim 7 wherein the ratio of the amount of non-anionic surfactants to the amount of anionic surfactant in the antimicrobial cleansing composition is less than 1:1.
9. An antimicrobial wipe effective against Gram positive bacteria, Gram negative bacteria, fungi, yeasts, molds, and viruses comprising a porous or absorbent sheet impregnated with an antimicrobial cleansing composition, wherein the antimicrobial cleansing composition comprises the following individual components:
  - a. from about 0.05% to about 1.0%, by weight of the antimicrobial cleansing composition, of an antimicrobial active;
  - b. from about 0.1% to about 2%, by weight of the antimicrobial cleansing composition, of an anionic surfactant;
  - c. from about 0.5% to about 8%, by weight of the antimicrobial cleansing composition, of a proton donating agent; and
  - d. from about 3% to about 99.85%, by weight of the antimicrobial cleansing composition, water;wherein the composition is adjusted to a pH of from about 3.0 to about 6.0;  
wherein the antimicrobial cleansing composition exhibits a One-wash Immediate Germ Reduction Index of greater than about 1.7; and wherein the antimicrobial composition has a Mildness Index of greater than 0.4.
10. An antimicrobial wipe according to claim 9 wherein the antimicrobial active is selected from the group consisting of triclosan, triclocarban, piroctone olamine, PCMX, ZPT, natural essential oils and their key ingredients, and mixtures thereof.
11. An antimicrobial wipe according to claim 10 wherein the antimicrobial active is triclosan.
12. An antimicrobial wipe according to claim 10 wherein the anionic surfactant has a Microtox Response Index of less than about 150.
13. An antimicrobial wipe according to claim 10 wherein the proton donating agent is an organic acid having a Buffering Capacity of greater than about 0.005.
14. An antimicrobial wipe according to claim 10 wherein the proton donating agent is a mineral acid.
15. An antimicrobial wipe according to claim 13 wherein the composition is adjusted to a pH of from about 3.5 to about 5.0.
16. An antimicrobial wipe according to claim 15 wherein the ratio of the amount of non-anionic surfactants to the amount of anionic surfactant in the antimicrobial cleansing composition is less than 1:1.
17. An antimicrobial wipe according to claim 16 which further comprises from about 1% to about 30%, by weight of the antimicrobial cleansing composition, of a lipophilic skin moisturizing agent.
18. A method for providing improved immediate reduction of germs on the skin comprising rubbing the antimicrobial wipe of claim 1 on human skin, wherein the antimicrobial cleansing composition is applied to the

skin in a safe and effective amount.

19. A method for providing improved immediate reduction of germs on the skin, comprising rubbing the antimicrobial wipe of claim 9 on human skin, wherein the antimicrobial cleansing composition is applied to the skin in a safe and effective amount.

20. A method for treating acne, comprising rubbing the antimicrobial wipe of claim 1 on human skin, wherein the antimicrobial cleansing composition is applied to the skin in a safe and effective amount.

21. An antimicrobial wipe according to claim 1 wherein the antimicrobial cleansing composition comprises from about 0.05% to about 5%, by weight of the antimicrobial cleansing composition, of the anionic surfactant.

22. An antimicrobial wipe according to claim 1 wherein the antimicrobial cleansing composition comprises from about 0.05% to about 2%, by weight of the antimicrobial cleansing composition, of the anionic surfactant.

23. An antimicrobial wipe according to claim 1 wherein the ratio of the amount of non-anionic surfactants to the amount of anionic surfactant in the antimicrobial cleansing composition is less than 1:2.

24. An antimicrobial wipe according to claim 1 wherein the ratio of the amount of non-anionic surfactants to the amount of anionic surfactant in the antimicrobial cleansing composition is less than 1:4.



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L8: Entry 5 of 7

File: USPT

Nov 11, 1997

DOCUMENT-IDENTIFIER: US 5686088!A

TITLE: Antimicrobial wipe compositions**Abstract Paragraph Left (1):**

The present invention relates to improved topical pharmaceutical compositions in the form of a wipe or towelette having improved aesthetics which are useful for imparting antimicrobial actives. In particular, it relates to topical moisturizing wipe compositions containing one or more antimicrobial active compounds. These compositions are also useful for providing relief from symptoms associated with, for example, vaginal yeast infections.

**Brief Summary Paragraph Right (17):**

The compositions of the instant invention comprise one or more water-soluble humectants/moisturizers. A variety of humectants/moisturizers can be employed and can be present at a level of from about 1% to about 10%, more preferably from about 2% to about 8% and most preferably from about 3% to about 5%. These materials include the C.sub.3 -C.sub.6 diols and triols; urea; guanidine; glycolic acid and glycolate salts (e.g. ammonium and quaternary alkyl ammonium); lactic acid and lactate salts (e.g. ammonium and quaternary alkyl ammonium); polyhydroxy alcohols such as sorbitol, glycerin, hexanetriol, propylene glycol, hexylene glycol and the like; polyethylene glycol; sugars and starches; sugar and starch derivatives (e.g. alkoxylated glucose); D-panthenol; hyaluronic acid; lactamide monoethanolamine; acetamide monoethanolamine; and mixtures thereof.

**Brief Summary Paragraph Right (29):**

In highly preferred embodiment, the compositions are substantially free of materials which are insoluble or not colloiddally soluble in distilled water at 20.degree. C. Such materials include many conventional emollient materials such as hydrocarbon oils and waxes, fatty alcohols, certain fatty alcohol ethers and sterols extracted from lanolin, beeswax derivatives, vegetable waxes, sterols and amides. The compositions can, however, contain low levels of insoluble ingredients added, for example for visual effect purposes, e.g., titaniumated mica.

**Brief Summary Paragraph Right (31):**

The compositions of the invention have no need of additional surfactant materials which are conventionally added to cosmetic cream and lotion compositions in order to emulsify a water-insoluble oily phase.

**Brief Summary Paragraph Right (34):**

The compositions which are in aqueous form are also preferably substantially free of oil, i.e. contain less than about 1%, and preferably less than about 0.1% of materials which are insoluble or which are not colloiddally-soluble in the aqueous gel matrix at 10.degree. C. "Colloiddally-soluble" herein refers to particles in the usual colloiddal size range, typically from 1 to 1000 nm, especially from 1 to 500 nm. In highly preferred embodiment, the compositions are substantially free of materials which are insoluble or not colloiddally soluble in distilled water at 20.degree. C. Such materials include many conventional emollient materials such as hydrocarbon oils and waxes, fatty alcohols, certain fatty alcohol ethers and sterols extracted from lanolin, beeswax derivatives, vegetable waxes, sterols and amides. The compositions are also preferably substantially free of alcohol, i.e. contain less than about 5%, preferably less than about 0.3%, more preferably less than about 1% and most preferably less than about 0.5% alcohol. The compositions can, however, contain low levels of insoluble ingredients added, for example for visual effect purposes, e.g., titaniumated mica.

**Brief Summary Paragraph Right (36):**

The compositions of the present invention preferably comprise at least one emollient. Useful emollients have a required HLB below about 10. Preferred emollients are volatile silicone oils, non-volatile emollients, and the highly branched hydrocarbons known as the Permethyl 99 through 108A series (available from Permethyl

Corporation) and mixtures thereof. The compositions of the present invention more preferably comprise at least one volatile silicone oil which functions as a liquid emollient, or especially in a mixture of volatile silicone oils and non-volatile emollients. The term "volatile", as used herein, refers to those materials which have a measurable vapor pressure at ambient temperature.

**Brief Summary Paragraph Right (37):**

Volatile silicone oils useful in the compositions of the present invention are preferably cyclic. The following formula illustrates cyclic volatile polydimethylsiloxanes useful in the compositions disclosed herein: ##STR1## wherein n equals about 3 to about 7. Linear polydimethylsiloxanes contain from about 3 to about 9 silicon atoms per molecule and have the following general formula:

**Brief Summary Paragraph Right (38):**

Examples of preferred volatile silicone oils useful herein include: Dow Corning 344, Dow Corning 345, and Dow Corning 200 (manufactured by Dow Corning Corp.); Silicone 7207 and Silicone 7158 (manufactured by the Union Carbide Corp.); SF 1202 (manufactured by General Electric); and SWS-03314 (manufactured by SWS Silicones, Inc.).

**Brief Summary Paragraph Right (39):**

The present compositions also preferably contain one or more non-volatile emollients. Such materials include fatty acid and fatty alcohol esters, hydrocarbons, non-volatile silicone oils, and mixtures thereof. Emollients among those useful herein are described in 1 Cosmetics, Science and Technology, 27-104 (M. Balsam and E. Sagatin, Ed.; 1972), and U.S. Pat. No. 4,202,879, to Shelton, issued May 13, 1980 (both incorporated by reference herein).

**Brief Summary Paragraph Right (40):**

Non-volatile silicone oils useful as an emollient material include polyalkylsiloxanes and polyalkylarylsiloxanes. The essentially non-volatile polyalkyl siloxanes useful herein include, for example, polydimethyl siloxanes with viscosities of from about 5 to about 100,000 centistokes at 25.degree. C. Among the preferred non-volatile emollients useful in the present compositions are the polydimethyl siloxanes having viscosities from about 10 to about 400 centistokes at 25.degree. C. Such polyalkyl siloxanes include the Vicasil series (sold by General Electric Company) and the Dow Corning 200 series (sold by Dow Corning Corporation). Polyalkylaryl siloxanes include poly methylphenyl siloxanes having viscosities of from about 15 to about 65 centistokes at 25.degree. C. These are available, for example, as SF 1075 methylphenyl fluid (sold by General Electric Company) and 556 Cosmetic Grade Fluid (sold by Dow Corning Corporation).

**Brief Summary Paragraph Right (41):**

Non-polar fatty acid and fatty alcohol esters useful herein as an emollient material include, for example, ethyl hexyl palmitate, isodecyl neopentanoate, octadodecyl benzoate, diethyl hexyl maleate and PPG-2 myristyl ether propionate. Hydrocarbons such as isohexadecane (e.g., Permethyl 101A supplied by Presperse), petrolatum and USP light (e.g. Klearol.RTM.) or heavy (e.g. Kaydol.RTM.) mineral oils are also useful as emollients. Emollients useful in the instant invention are further described in U.S. Pat. No. 4,919,934, to Deckner et al., issued Apr. 24 1990, which is incorporated herein by reference in its entirety.

**Brief Summary Paragraph Right (44):**

The composition may also comprise from about 0.1% to about 2% of a thickening agent. Examples of suitable thickening agents include: cellulose derivatives (e.g., methyl cellulose and hydroxy propylmethyl cellulose), synthetic high molecular weight polymers (e.g., carboxyvinyl polymer and polyvinyl alcohol), plant hydrocolloids (e.g., karaya gum and tragacanth gum), clay thickeners (e.g., colloidal magnesium aluminum silicate and bentonite), and carboxyvinyl polymers are described in detail in U.S. Pat. No. 2,798,053, Brown, issued Jul. 2, 1975, incorporated herein by reference). A more complete disclosure of thickening agent useful herein can be found in Segarin, Cosmetics, Science and Technology, 2nd Edition, Vol. 1, pp. 72-73 (1972), incorporated herein by reference.

**Brief Summary Paragraph Right (55):**

Useful anesthetic or antipruritic drugs are selected from the group consisting of lidocaine, lidocaine hydrochloride, bupivacaine hydrochloride, chlorprocaine hydrochloride, dibucaine hydrochloride, etidocaine hydrochloride, mepivacaine hydrochloride, tetracaine, tetracaine hydrochloride, dyclonine hydrochloride and hexylcaine hydrochloride, benzocaine, benzyl alcohol, butamben picrate, camphor, camphorated metacresol, dibucaine, dibucaine hydrochloride, dimethisoquin hydrochloride, diphenhydramine hydrochloride, juniper tar, menthol, phenol, phenolate sodium, pramoxine hydrochloride, resorcinol and mixtures thereof.

**Brief Summary Paragraph Right (64):**

Other textile-length fibers of a synthetic or man-made origin may be used in various proportions to replace either partially or perhaps even entirely the previously-named fibers. Such other fibers include: polyamide fibers such as nylon 6, nylon 66, nylon 610, etc.; polyester fibers such as "Dacron", "Fortrel" and "Kodel"; acrylic fibers such as "Acrilan", "Orlon" and "Creslan"; modacrylic fibers derived from polyethylene and polypropylene; cellulose ester fibers such as "Arnel" and "Acele"; polyvinyl alcohol fibers, etc.

**Brief Summary Paragraph Right (67):**

The resulting fibrous web or sheet, regardless of its method of production, is then subjected to at least one of several types of bonding operations to anchor the individual fibers together to form a self-sustaining web incorporating the resins described infra. One method is to impregnate the fibrous web over its entire surface area with the resins of the present invention. Such over-all impregnation produces a nonwoven fabric of good longitudinal and cross strength, acceptable durability and solvent resistable and satisfactory abrasion resistance.

**Brief Summary Paragraph Left (2):**

wherein n equals about 1 to about 7. Linear volatile silicone materials generally have viscosities of less than about 5 centistokes at 25.degree. C. while cyclic materials typically have viscosities of less than about 10 centistokes. A description of various volatile silicone oils is found in Todd, et al., "Volatile Silicone Fluids for Cosmetics", Cosmetics & Toiletries, 91, pages 27-32 (1976), the disclosures of which are incorporated by reference herein in their entirety.

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L8: Entry 6 of 7

File: USPT

Apr 2, 1991

DOCUMENT-IDENTIFIER: US 5004643 A

TITLE: Silicone polymer-internally coated websAbstract Paragraph Left (1):

An improved process is provided for treating a porous web (especially fabric) to produce a novel silicone polymer internally coated web. In the process, a starting curable liquid silicone polymer is coated under pressure upon one surface of the web, and the web is then subjected to localized shear forces sufficient to move the silicone polymer composition into interior portions of the web and to distribute the silicone polymer composition generally uniformly therewithin in such planar region. Excess silicone polymer composition is wiped away from a web surface. Thereafter, the resulting web is heated or irradiated to cure the silicone polymer. Preferably a web is preliminarily impregnated with a fluorochemical. Webs procuded by this process are breathable, waterproof or highly water repellent, and flexible.

Brief Summary Paragraph Right (2):

The present invention generally concerns porous fibrous webs (especially fabrics) that are modified in their properties by incorporation therein to an internal layer of silicone polymer. Such webs are prepared by pressurized impregnation methods.

Brief Summary Paragraph Right (4):

In the prior art, it has been proposed to treat porous webs, especially fabrics, with silicone resins and also with fluorochemicals.

Brief Summary Paragraph Right (6):

For example, U.S. Pat. Nos. 3,436,366; 3,639,155; 4,472,470; 4,500,584; and 4,666,765 disclose silicone coated fabrics. Silicone coatings are known to exhibit relative inertness to extreme temperatures of both heat and cold and to be relatively resistant to ozone and ultraviolet light. Also, a silicone coating can selectively exhibit resistance to soiling, strength enhancement, and/or flame retardancy.

Brief Summary Paragraph Right (8):

Prior art fluorochemical and silicone fabric treatment evidently each can protect only that side of the fabric upon which they are disposed. Such treatments characteristically significantly alter the hand, or tactile feel, of the treated side. Prior silicone fabric coatings typically degrade the tactile finish, or hand, of the fabric and give the coated fabric side a rubberized finish which is not appealing for many fabric uses, particularly garments.

Brief Summary Paragraph Right (13):

Porous webs have been further shown to be surface coated in, for example, U.S. Pat. Nos. 4,478,895; 4,112,179; 4,297,265; 2,893,962; 4,504,549; 3,360,394; 4,293,611; 4,472,470; and 4,666,765. These surface coatings impart various characteristics to the surface of a web, but do not substantially impregnate the web fibers. Such coatings remain on the surface and do not provide a film over the individual internal fibers and/or yarn bundles of the web. In addition, such coatings on the web surface tend to wash away quickly.

Brief Summary Paragraph Right (15):

In order to treat a flexible web, by heavy saturation, or impregnation, with a polymer material, such as a silicone resin, the prior art has suggested immersion of the flexible web, or fabric, in a padbath, or the like, using a low viscosity liquid silicone resin so that the low viscosity liquid can flow readily into, and be adsorbed or absorbed therewithin. The silicone resin treated product is typically a rubberized web, or fabric, that is very heavily impregnated with silicone. Such a treated web is substantially devoid of its original tactile and visual properties, and instead has the characteristic rubbery properties of a cured silicone polymer.

**Brief Summary Paragraph Right (18):**

Coating at a predetermined thickness can be achieved by using precision-controlled deposition of coating material followed by passage through a pair of opposed scraping knives. The knives smooth the coating and maintain the thickness of the coating to a desired thickness. For example, it is possible to apply a relatively thick silicone liquid elastomer coating to a rough web, typically of fiberglass, in order to make architectural fabric as is taught in U.S. Pat. No. 4,666,765. In this example, the drag knives are set to a thickness of about 2 to 10 mils thicker than the web thickness. This setting, depending on the coating speed, can yield a base coat thickness of approximately 3 to 12 mils thicker than the web thickness.

**Brief Summary Paragraph Right (20):**

One prior art silicone resin composition is taught by U.S. Pat. Nos. 4,472,470 and 4,500,584, and includes a vinyl terminated polysiloxane, typically one having a viscosity of up to about 2,000,000 centipoises at 25.degree. C., and a resinous organosiloxane polymer. The composition further includes a platinum catalyst, and an organohydrogenpolysiloxane crosslinking agent, and is typically liquid. Such composition is curable at temperatures ranging from room temperature to 100.degree. C. or higher depending upon such variables as the amount of platinum catalyst present in the composition, and the time and the temperature allowed for curing.

**Brief Summary Paragraph Right (21):**

Such compositions may additionally include fillers, including finely divided inorganic fillers. Silicone resin compositions that are free of any fillers are generally transparent or translucent, whereas silicone resin compositions containing fillers are translucent or opaque depending upon the particular filler employed. Cured silicone resin compositions are variously more resinous, or hard, dependent upon such variables as the ratio of resinous copolymer to vinyl terminated polysiloxane, the viscosity of the polysiloxane, and the like.

**Brief Summary Paragraph Right (23):**

This invention relates to a flexible porous web which contains an internal coating of a silicone polymer composition.

**Brief Summary Paragraph Right (24):**

The silicone polymer composition has a viscosity that is sufficient to achieve an internal coating of the web. Generally, the viscosity is greater than about 1000 centipoise and less than about 2,000,000 centipoise. Such composition, when cured, is preferably elastomeric.

**Brief Summary Paragraph Right (25):**

Preferably, a fluorochemical is impregnated into the web before the silicone polymer is applied.

**Brief Summary Paragraph Right (26):**

In a web of this invention the quantity of silicone polymer can vary widely. The silicone polymer composition is present in an amount that is sufficient to achieve an internal coating of the web. Generally, this amount is in the range of about 5 to about 200 weight percent of the weight of the untreated web or fibers. When present, the quantity of fluorochemical is in the range of about 0.01 to about 5 weight percent of the weight of the untreated web or fibers. When, as is preferred, a web incorporates both a fluorochemical and a silicone polymer, they are present in an amount sufficient to achieve an internal coating of the web. Generally, the total weight of fluorochemical and silicone polymer is in the range of about 5 to about 200 weight percent of the weight of the untreated web.

**Brief Summary Paragraph Right (27):**

Notwithstanding the large amount of silicone present in webs of the present invention, they surprisingly retain porosity, breathability, flexibility, hand and other characteristics similar to untreated webs while exhibiting improved characteristics such as water repellancy, rewashability, service life, abrasion resistance and durability.

**Brief Summary Paragraph Right (31):**

Webs of the present invention contain a curable silicone polymer impregnant that is present as a film, or coating, or layer within a web that envelopes at least a portion of the fibers or cell or pore walls of the web. The interstices or open cells in the region of the internal coating are mostly filled or plugged by impregnant. The outer surfaces of the web are preferably substantially free of impregnant. However, the web remains breathable and is either water resistant or waterproof. The thickness of the film, coating or layer is generally in the range of 0.01 to 50 microns.

**Brief Summary Paragraph Right (33):**

Silicone polymer which substantially, completely encapsulates a web's fibers or lines its cell or pore walls and forms an internal layer means, that the silicone polymer is located mostly upon surface portions of the interior of the web.

**Brief Summary Paragraph Right (34):**

Depending upon the conditions used to produce it, a web produced in accordance with the present invention can characteristically and preferably exhibit a soft hand and flexibility that is comparable to the hand and flexibility of the untreated web. In some cases, the difference in hand between the treated and untreated webs may not be perceptible. This is particularly surprising in view of the substantial amount of silicone polymer being added to the web. A treated web has a breathability which, by a present preference, can approach that of the untreated web notwithstanding the relatively large amount of silicone polymer present.

**Brief Summary Paragraph Right (35):**

A silicone polymer composition having a viscosity in the range above indicated is used to produce the treated webs. If desired, additives can be admixed with such a composition to adjust and improve properties of such composition or web, such as viscosity and/or rheology, combustibility, reflectivity, flexibility, conductivity, light fastness, mildew resistance, rot resistance, stain resistance, grease resistance, and the like. In general, a web of this invention exhibits enhanced durability.

**Brief Summary Paragraph Right (38):**

The silicone polymer composition prolongs the use and service life of a web, usually by at least an order of magnitude, depending on such factors as web type, extent and type of treatment by the teachings of this invention, and the like.

**Brief Summary Paragraph Right (39):**

Optionally, and as indicated above, agents or additives carried by the silicone polymer composition into a web can be stably fixed in the web with the cured silicone polymer. For example, agents such as ultraviolet light absorbers, dulling agents, reflectivity enhancers, and the like, which modify a web's response to light and radiation are desirably located substantially upon the surfaces of the web's fibers. When these agents are incorporated into the enveloping silicone polymer film, it appears that then they are retained where they are deposited.

**Brief Summary Paragraph Right (40):**

A present preference in the practice of this invention is to employ a silicone polymer composition that contains a benzophenone.

**Brief Summary Paragraph Right (41):**

In addition, the present invention is directed to processes for making silicone polymer internally coated webs. Such processes involve tensioning a porous, flexible web, applying a curable silicone polymer composition thereto, and then moving a generally uniformly applied localized shear force over and against one surface of the tensioned web. The shear force is sufficient to shear thin the silicone polymer, to distribute the silicone polymer composition within the web as an internal coating in a region extending generally in spaced, parallel relationship to at least one face of the web and to generally envelop surface portions of at least some of the web fibers or form a lining of the cells or pores of the web. The web is then optionally interveningly stored, or is (preferably) immediately subjected to curing conditions (heat, moisture and/or radiation) which converts the polymer composition as deposited in the web into a solid elastomeric polymer.

**Drawing Description Paragraph Right (2):**

FIG. 1 is a graphical plot illustrating the flow of the silicone polymer composition over time upon and in fabrics both pretreated and untreated with water repellent chemicals, such as fluorochemicals;

**Drawing Description Paragraph Right (3):**

FIG. 2 is a plan view of a prior art silicone polymer treated fabric magnified 150 times;

**Drawing Description Paragraph Right (6):**

FIG. 3c is a view of the side of the fabric of FIG. 3a that is the opposite of the side to which silicone polymer was applied;

**Detailed Description Paragraph Right (18):**

Synthetic leathers which are coated fabrics, like poromerics, have a balance of physical properties and economic considerations. Usually the coating is either vinyl or urethane. Vinyl coatings can be either solid or expanded vinyl which has internal air bubbles which are usually a closed-cell type of foam. Because such structures usually have a non-porous exterior or front surface or face, such structures display poor breathability and moisture vapor transmission. However, since the interior or back surface or face is porous, such a coated fabric can be used in the practice of this invention by applying the impregnant silicone polymer to the back fact.

Detailed Description Paragraph Right (20):

The term "impregnation", or "impregnate", as used herein, refers to forcing a liquid substance into a porous solid, such as a flexible web or substrate.

Detailed Description Paragraph Right (22):

With respect to the silicone polymer compositions used in this invention, the term "impregnation" refers to the penetration of such polymeric composition into a porous web, to the distribution of such composition in a controlled manner through such web, and to the resultant, at least partial envelopment of at least a portion of the fibers of such web by such composition in accordance with the present invention.

Detailed Description Paragraph Right (26):

The term "elastomeric" as used herein refers to the ability of a cured silicone polymer impregnated web to stretch and return to its original state.

Detailed Description Paragraph Right (27):

A characteristic of a silicone polymer composition, or impregnant, that is used for impregnation into a web or substrate in accordance with the teachings of this invention is that apparently only a minimum quantity of the impregnant appears actually to enter into the fibers, cells or pores comprising such web or substrate. The exact amount of impregnant which enters into individual fibers, cells or pores is unknown, but is now estimated to be typically below about 10 weight percent of the total quantity of impregnant applied to a web or substrate, with the remainder of the impregnant appearing to comprise mainly deposits on and around fibers, cells or pores of a web.

Detailed Description Paragraph Right (28):

The term "curing", or "cure", as used herein, refers to a change in state, condition, and/or structure in a material, such as a curable silicone polymer composition that is usually, but not necessarily, induced by at least one applied variable, such as time, temperature, radiation, presence and quantity in such material of a curing catalyst or curing accelerator, or the like. In the occurrence of curing in any case, such as the curing of such a polymer composition that has been impregnated into a porous flexible substrate or web, the components of such a composition may experience occurrence of one or more of complete or partial (a) polymerization, (b) cross-linking, or (c) other reaction, depending upon the nature of the composition being cured, application variables, and presumably other factors.

Detailed Description Paragraph Right (29):

The term "filled" as used herein in relation to interstices or open cells, and to the amount of silicone polymer composition therein in a given web or substrate, designates the presence of such composition therein. When a given interstice or open cell is totally taken up by such composition, it is "completely filled" or "plugged".

Detailed Description Paragraph Right (31):

A flexible, porous fibrous web is preferably untreated or scoured before being treated in accordance with the present invention. Preferably a web is preliminarily treated, preferably saturated, for example, by padding, to substantially uniformly impregnate the web with a fluorochemical. Typically, and preferably, the treating composition comprises a dispersion of fluorochemical in a liquid carrier. The liquid carrier is preferably aqueous and can be driven off with heat after application. The treating composition has a low viscosity, typically comparable to the viscosity of water or less. After such a treatment, it is presently preferred that the resulting treated web exhibits a contact angle with water measured on an outer surface of the treated web that is greater than about 90 degrees. The treated web preferably contains fluorochemical substantially uniformly distributed therethrough. Thus, the fluorochemical is believed to be located primarily on and in the individual fibers, cells or pores with the web interstices or open cells being substantially free of fluorochemical.

Detailed Description Paragraph Right (32):

A presently preferred concentration of fluorochemical in a treatment composition is typically in the range of about 1 to about 10% fluorochemical by weight of the total treating composition weight, and more preferably is

about 2.5% of an aqueous treating dispersion. Web weight add-ons of the fluorochemical can vary depending upon such factors as the particular web treated, the silicone polymer impregnant to be utilized in the next step of the treatment process of this invention, the ultimate intended use and properties of the treated web of this invention, and the like. The fluorochemical weight add-on is typically in the range of about 0.01 to about 5% of the weight of the untreated web. After fluorochemical impregnation, the web is preferably squeezed to remove excess fluorochemical composition after which the web is heated or otherwise dried to evaporate carrier liquid and thereby also accomplish fluorochemical insolubilization or sintering, if permitted or possible with the particular composition used.

Detailed Description Paragraph Right (33):

The fluorochemical treated web is thereafter impregnated under pressure by the procedures taught by this invention, with a predetermined amount of a curable silicone polymer impregnant composition to form a web whose fibers, cells or pores are at least partially enveloped or lined with the curable silicone polymer impregnant, whose web outer surfaces are substantially free of the curable impregnant, whose web interstices or open cells are not completely filled with the curable impregnant and which contains an internal layer of silicone polymer. The curable impregnant composition utilized preferably exhibits a viscosity greater than 1,000 centipoise and less than 2,000,000 centipoise at rest at 25.degree. C. at a shear rate of 10 reciprocal seconds.

Detailed Description Paragraph Right (34):

The silicone polymer impregnant composition can include conventional additives.

Detailed Description Paragraph Right (35):

The fluorochemical residue that remains after fiber treatment may not be exactly evenly distributed throughout the web, but may be present in the web in certain discontinuities. For example, these discontinuities may be randomly distributed small areas upon an individual fiber's surface. However, the quantity and distribution of fluorochemical through a web is believed to be largely controllable. Some portions of the fluorochemical may become dislodged from the web and migrate through the silicone polymer due to the forces incurred by cause the shear thinning of the silicone polymer.

Detailed Description Paragraph Right (36):

The curable silicone impregnant composition is believed to be typically polymeric, to be usually a mixture of co-curable polymers and oligomers, and to include a catalyst to promote the cure.

Detailed Description Paragraph Right (37):

The silicone polymer impregnant composition can include, as additive components, polyurethanes, fluorosilicones, silicone-modified polyurethanes, acrylics, polytetrafluoroethylene-containing materials, and the like.

Detailed Description Paragraph Right (39):

It is to be understood that the depth of silicone polymer impregnation into a web can be controlled by the application procedures herein described to provide a selective placement of the silicone polymer impregnant within the substrate or web. This allows the shear thinning, i.e., viscosity reduction, action to take place throughout the web.

Detailed Description Paragraph Right (40):

The silicone polymer composition is theorized to be caused to flow and distribute itself over fibers, cells or pores in a web under the influence of the processing conditions provided by this invention. This flow and distribution is further theorized to be facilitated and promoted by the presence of a fluorochemical which has been preliminarily impregnated into a web, as taught herein. The amount of fluorochemical or fluorochemical residue in a web is believed to influence the amount, and the locations, where the liquid silicone polymer impregnant will collect and deposit, and produce an internal coating in the web. However, there is no intent to be bound herein by theory.

Detailed Description Paragraph Right (41):

Some portion of the residue of fluorochemical resulting from a preliminary web saturating operation is theorized to be present upon a treated fiber's surfaces after envelopment of fibers, cells or pores by the silicone polymer has been achieved during internal web coating by the practice of this invention. This is believed to be demonstrated by the fact that a web of this invention still exhibits an enhanced water and oil repellency, such as is typical of fluorochemicals in porous webs. It is therefore believed that the fluorochemicals are affecting the adherence of the silicone polymer as a thin film enveloping layer about the treated fibers, cells or pores as well



as facilitating liquid silicone polymer impregnant pressurized flow within and about the interstices or open cells of the web being treated so that the silicone can assume its position enveloping the fibers or lining the cells or pores of the substrate.

Detailed Description Paragraph Right (42):

The exact interrelationship between the silicone polymer film and the impregnated fluorochemical is presently difficult, or perhaps impossible, to quantify because of the variables involved and because transparent silicone polymer is difficult to observe by optical microscopy. It can be theorized that perhaps the silicone polymer and the fluorochemical each tend to produce discontinuous films upon fiber surface, and that such films are discontinuous in a complementary manner. It may alternatively be theorized that perhaps the silicone polymer film is contiguous, or substantially so, relative to fluorochemical molecules on a fiber surface, and that the layer of silicone polymer on a fiber surface is so thin that any dislodgement of the fluorochemical may release the fluorochemical into the silicone polymer film thereby allowing the fluorochemical to orient with the required cure temperature of the silicone, reactivating the water surface contact angle so that the water repellant properties of an underlying fluorochemical are exorable through the silicone polymer film. However, regardless of physical or chemical explanation, the combination of silicone polymer film and fluorochemical results in a fiber envelopment or cell or pore wall lining and the formation of an internal layer of silicone polymer in a web when this invention is practiced. After curing, the silicone polymer is permanently fixed material.

Detailed Description Paragraph Right (43):

By using the impregnation method provided by this invention, one can achieve an impregnation of a silicone polymer composition into a porous substrate or web to obtain a desired treated web.

Detailed Description Paragraph Right (44):

A curable silicone polymer such as used in the practice of this invention is applied under pressure using shear forces onto and into a web or substrate. The shear forces cause the curable silicone polymer to flow into the web. The extent of fiber envelopment and cell or pore wall lining is believed to be regulatable by controlling such factors as the selection and applied amount of fluorochemical and curable silicone polymer in combination with the applied compressive and shear forces employed at a given temperature so that fiber envelopment is achieved while the interstices and/or open cells of the web are not completely filled with such polymer in the region of the internal coating, and the outer opposed surfaces of the web are substantially completely free of silicone polymer coating or residue. After such an impregnation procedure, the curable silicone impregnant is then cured.

Detailed Description Paragraph Right (45):

The curable silicone polymer impregnant is applied in an amount at least sufficient to partially saturate the web and fill some of the interstices or open cells of the web. Then, the web, while tensioned, is passed over and against shearing means or through a compression zone, such as between rollers or against a shear knife. Thus transversely applied shear force and compressive pressure is applied to the web. The combination of tensioning, shearing forces, and web traveling speed is sufficient to cause the curable silicone polymer impregnant to move into the web, out from the interstices or open cells around the web fibers, cells or pores being enveloped, thereby leaving at least some of the interstices and/or open cells unfilled in regions of the web outside of the region occupied by the interior coating, and preferably substantially free of, silicone polymer impregnant. Excess silicone polymer is removed by the surface wiping action of the shearing means. The curable silicone polymer impregnant enveloping the fibers is thereafter cured.

Detailed Description Paragraph Right (46):

The desired impregnation or penetration of, and distribution of silicone polymer in, a web is believed to be achieved by localized pressuring forces exerted on a web surface which are sufficiently high to cause the viscosity of a silicone polymer impregnant composition to be locally reduced, thereby permitting such silicone impregnant to flow under such pressuring and to impregnate the web and to envelope its fibers or line the cell or pore walls thereof. To aid in this process, the web is preferably at least slightly distorted by tensioning or stretching, while being somewhat transversely compressed at the location of the impregnation. This distortion is believed to facilitate the entrance of the silicone polymer composition into the web. When the compression and tension are released, the silicone polymer composition is believed to be squeezed or compressed within and through the interstitial spaces, or open cell spaces, of the treated web.

Detailed Description Paragraph Right (47):

If, for example, too much silicone polymer is present in the finished product, then either or both the tension and shear force can be increased, and vice versa for too little silicone polymer. If flow is not adequate upon the

fibers, producing incomplete fiber envelopment, then the viscosity of the silicone impregnant composition can be reduced by reducing the pressures and temperatures employed for the impregnation. Alternatively, if the viscosity is excessive, then the impregnating pressure and/or temperature can be increased. Opposite adjustments should be made if silicone polymer impregnant flow is insufficiently viscous. If the silicone polymer impregnating composition is resistant to being positioned in a desired location in a desired amount in a given web at various viscosities and/or pressures, then the level of fluorochemical pretreatment of the web can be increased, as in the case of overimpregnation, or decreased, as in the case of underimpregnation.

Detailed Description Paragraph Right (48):

In one embodiment of an impregnation procedure, the pressured impregnation of a web occurs between two rollers. One such roller bears a silicone polymer impregnant, typically and preferably distributed uniformly upon and over a circumferentially extending textured, or gravure surface. Such roller rotates (i) in the same direction as a facing roller and (ii) oppositely to the direction of movement of a continuously moving web traveling past the localized impregnation area achieved between such roller and such moving web. The unidirectional rotation of the two rollers is believed to produce a distorting and stretching force or effect upon the web. This force is believed to promote penetration of the silicone polymer impregnant into the web. This form of impregnant application or coating can be termed "reverse roll coating" for convenience. Preferably, the reverse coating rollers have generally horizontal axes while the moving web moves generally horizontally. The web is further concurrently both longitudinally tensioned and distorted by being stretched against metering bars, bar knives, and the like which are urged against the web.

Detailed Description Paragraph Right (49):

Such an initial pressured impregnation step is preferably followed by a series of further pressured web treatment steps believed to accomplish impregnant reintroduction, impregnant distribution, impregnant scraping, and excess impregnant removal and recovery. The collective result of such steps gradually produces a web wherein the silicone polymer impregnant envelopes to a desired extent the fibers or lines the cell or pore walls comprising the web and collects within a desired internal region or zone in the web thereby filling or plugging interstitial spaces, or open cells or pores, of the web in such region, but not filling the internal structure of the treated web with silicone polymer beyond a desired extent. Particularly, and for example, in a fabric, a silicone polymer composition may be made to substantially completely envelope the fibers or line the cells or pores thereof and fill the interstitial spaces thereof in such internal region.

Detailed Description Paragraph Right (50):

In another embodiment of an impregnation procedure, application of silicone polymer impregnant to a web occurs from a reservoir. This reservoir of silicone impregnant is positioned tightly against the tensioned, moving web (or fabric). The linearly extending, preferably vertically upwardly moving, web (or fabric), constitutes a wall portion of the reservoir. Next, along the path of web travel, a bar or shear knife is pressed strongly and transversely against and laterally across the longitudinally tensioned web (or fabric). Further along the path of web movement, a shear blade or flexible scraper knife is also strongly and transversely forced laterally across and against the tensioned web. More than one shear knife, or more than one flexible compressive knife, can be successively positioned along the path of web movement. These blade means are believed to reintroduce the silicone impregnant into the web, to distribute the silicone polymer, and to promote and complete the envelopment of fibers or lining of the cell or pore walls and fillage of interstices and open cells with silicone polymer, and form an internal coating in a desired region in a web. These scraper knives or shear blades are also believed to force the silicone polymer impregnant further into the three-dimensional structure of the web. Also, these knives, particularly the scraper knives, wipe or scrape excess silicone polymer impregnant off the surface of the web, thereby regulating the amount of silicone polymer impregnated.

Detailed Description Paragraph Right (51):

The transversely applied shear forces applied across and against the web are sufficiently high to achieve temporarily and locally, a lowering of the viscosity of the preferably thixotropic viscous silicone polymer impregnant. The lowered viscosity silicone polymer impregnant is thus enabled to flow into, and upon, the internal three-dimensional structure of the web. Because the silicone polymer composition that is being applied is subject to cure with heat or radiation and time, and because the pressured impregnation is believed to produce localized heat, the shearing conditions used prior to curing are preferably controlled to minimize premature curing. The properties of the silicone polymer impregnant are preferably selected to be such that cure, or excessive cure, does not occur while the web is being treated with silicone polymer during the pressured impregnation. The cure preferably occurs only after the web impregnation procedure has been completed. Preferably, the cure temperature of the silicone polymer composition is relatively high (preferably above about 250.degree. F.) and the heat exposure time is such as is needed to obtain a desired solid resilient elastomeric

silicone polymer.

Detailed Description Paragraph Right (52):

If desired, the rheology of the silicone polymer impregnant may be altered or controlled. Characteristics of a web are believed to be influenced by rheology, but it is believed that, in general, this invention can be practiced without careful control of rheology while controlling viscosity.

Detailed Description Paragraph Right (53):

The viscosity of the silicone polymer impregnant is preferably lowered by the high pressure (shear) forces exerted during impregnation. However, such a pressure- and/or temperature-induced lowered viscosity should not go down too low, otherwise the impregnant can flow substantially uncontrolled in the web in the manner of a low viscosity liquid that is saturated and impregnated into a web as in prior art web treatments. If the viscosity of the silicone polymer composition is too low at the time of impregnation, then the web interstices or open cells can become excessively filled therewith, and the impregnant is not, for example, reliably and controllably applied to achieve an envelopment of the structural elements (including fibers) of the web being treated together with internal coating formation.

Detailed Description Paragraph Right (54):

Benzophenones, and particularly 2,4-dihydroxybenzophenone, are believed to be a particularly useful class of additives to the starting silicone polymer composition, as hereinbelow described.

Detailed Description Paragraph Right (55):

As above indicated, the activity transpiring at a final step in the practice of a method of this invention is generically referred to herein as curing. Conventional curing conditions known to the prior art for curing silicone polymer compositions are generally suitable for use in the practice of this invention. Thus, temperatures in the range of about 250.degree. F. to about 350.degree. F. are used and times in the range of about 30 seconds to about 1 minute can be used, although longer and shorter curing times and temperatures may be used, if desired, when thermal curing is practiced. Radiation curing, as with an electron beam or ultraviolet light can also be used. However, using platinum catalysts to accelerate the cure while using lower temperatures and shorter cure times is preferable.

Detailed Description Paragraph Right (58):

As indicated above, a web is preferably pretreated and impregnated with a fluorochemical prior to being impregnated under pressure with a silicone polymer composition as taught herein.

Detailed Description Paragraph Right (63):

Fluorochemicals are sometimes known in the art as durable water repellant (DWR) chemicals, although such materials are typically believed to be not particularly durable and to have a tendency to wash out from a fabric treated therewith. In contrast, fiber enveloped webs of this invention which have been pretreated with a fluorochemical display excellent durability and washability characteristics. Indeed, the combination of fluorochemical pretreatment and silicone polymer fiber envelopment such as provided by the present invention appears to provide synergistic property enhancement because the effects or properties obtained appear to be better than can be obtained than by using either the fluorochemical or the silicone polymer alone for web treatment.

Detailed Description Paragraph Right (80):

The web weight add-on provided by the fluorochemical after removal of volatiles is usually relatively minor. However, the weight add on can vary with such factors as the nature of web treated, the type of silicone impregnant utilized in the next step of the process, the temperature at which the impregnant is applied, the ultimate use contemplated for a web, and the like.

Detailed Description Paragraph Right (82):

Durability of a web that has been treated with a fluorochemical and durability of a web that is subsequently treated with a silicone polymer can sometimes be improved by the conventional process of "sintering". The exact physical and chemical processes that occur during sintering are unknown. The so-called sintering temperature utilized is a function of the fluorochemical composition utilized and such temperature is frequently recommended by fluorochemical manufacturers. Typically, sintering is carried out at a temperature of about 130.degree. to about 160.degree. C. for a period of time of about 2 to about 5 minutes. Acid catalysts can be added to give improved durability to laundering and dry cleaning solvents.

Detailed Description Paragraph Right (83):

The fluorochemical is believed to provide more than water or other repellent properties to the resulting treated (impregnated) web, particularly since the curable silicone impregnant is often itself a water repellent. Rather, and without wishing to be bound by theory, it is believed that the fluorochemical in a treated web provides relative lubricity for the treated fibers during the pressure application of the curable silicone polymer impregnant. The silicone polymer impregnant is applied under pressures which can be relatively high, and this impregnant is itself relatively viscous, as is discussed herein. In order for the curable silicone polymer impregnant to coat and envelope web fibers, but not fill web interstitial voids, the fibers of the web may move over and against each other to a limited extent, thereby to permit entry of the silicone impregnant into and around the fibers. It is thought that the fluorochemical deposits may facilitate such fiber motion and facilitate envelopment during the pressure impregnation and subsequent shearing processing.

Detailed Description Paragraph Right (84):

Alternatively, the fluorochemical may inhibit deposition of the silicone polymer impregnant at the positions of the fluorochemical deposits which somehow ultimately tends to cause thin enveloping layers of silicone polymer to form on fibers.

Detailed Description Paragraph Right (85):

The precise physics and chemistry of the interaction between the fluorochemical and the silicone impregnant is not understood. A simple experiment demonstrates movement of the liquid silicone polymer as influenced by the presence of the fluorochemical:

Detailed Description Paragraph Right (87):

A graphical plot of the flow of the silicone composition respectively upon the untreated and treated swatches over time can be prepared, such as shown in FIG. 1. At the expiration of 30 minutes the viscous composition has typically traveled a distance of about 8.8 centimeters upon the treated swatch, or a rate of about 0.29 centimeters per minute. At the expiration of the same 30 minutes, the viscous composition has typically traveled a lesser distance of about 7.1 centimeters upon the untreated swatch, or a rate of about 0.24 centimeters per minute. Qualitatively commensurate results are obtainable with other DWR fluorochemical adjuvants that facilitate the viscous flow of polymer compositions in accordance with the invention. Indeed, if desired, the simple flow rate test can be used to qualify an adjuvant compound for its employment within the method of the invention. The fluorochemical pretreated web generally increases the surface contact angle of the silicone polymer while reducing the amount of saturation of the silicone polymer into the fibers themselves.

Detailed Description Paragraph Right (88):

The fluorochemical treated web is thereafter impregnated under pressure with a predetermined amount of a curable silicone polymer impregnant composition to form a web whose fibers are preferably substantially completely enveloped with such curable impregnant and whose outer surfaces and interstices are preferably substantially completely free of the curable impregnant. The silicone polymer impregnant is thereafter cured by heat, radiation, or the like. Even room temperature curing can be used. A silicone polymer impregnated, fluorochemical pretreated web can be interveningly stored before being subjected to curing conditions depending upon the so-called pot life of the treating silicone polymer impregnant.

Detailed Description Paragraph Right (89):

A curable silicone polymer impregnant composition utilized in the practice of this invention preferably has a viscosity that is sufficient to achieve an internal coating of the web. Generally, the viscosity is greater than about 1000 centipoise and less than about 2,000,000 centipoise at a shear rate of 10 reciprocal seconds. It is presently most preferred that such composition have a viscosity in the range of about 5,000 to about 10,000 centipoise at 25.degree. C. Such a composition is believed to contain less than about 1% by weight of volatile material.

Detailed Description Paragraph Right (90):

The silicone polymer is believed to be typically polymeric and to be commonly a mixture of co-curable polymers, oligomers, and/or monomers. A catalyst is usually also present, and, for the presently preferred silicone polymer compositions discussed hereinafter, is platinum or a platinum compound, such as a platinum salt.

Detailed Description Paragraph Right (91):

A preferred class of liquid curable silicone polymer compositions comprises a curable mixture of the following components:

Detailed Description Paragraph Right (96):

Typical silicone hydrides (component A) are polymethylhydrosiloxanes which are dimethyl siloxane copolymers. Typical vinyl terminated siloxanes are vinyl dimethyl terminated or vinyl substituted polydimethylsiloxanes. Typical catalyst systems include solutions or complexes of chloroplatinic acid in alcohols, ethers, divinylsiloxanes, and cyclic vinyl siloxanes.

Detailed Description Paragraph Right (97):

The polymethylhydrosiloxanes (component A) are used in the form of their dimethyl copolymers because their reactivity is more controllable than that of the homopolymers and because they result in tougher polymers with a lower cross-link density. Although the reaction with vinyl functional silicones (component B) does reportedly take place in 1:1 stoichiometry, the minimum ratio of hydride (component A) to vinyl (component B) in commercial products is reportedly about 2:1 and may be as high as 6:1. While the hydrosilation reaction of polymethylhydrosilane is used in both so called RTV (room temperature vulcanizable) and LTV (low temperature vulcanizable) systems, and while both such systems are believed to be useful in the practice of the present invention, systems which undergo curing at elevated temperature are presently preferred.

Detailed Description Paragraph Right (99):

Particulate fillers are known to be useful additives for incorporation into liquid silicone polymer compositions. Such fillers apparently not only can extend and reinforce the cured compositions produced therefrom, but also can favorably influence thixotropic behavior in such compositions. Thixotropic behavior is presently preferred in compositions used in the practice of this invention. A terminal silanol (Si-OH) group makes such silanol siloxanes susceptible to reaction in curing, as is believed desirable.

Detailed Description Paragraph Right (101):

A silicone composition useful in this invention can contain curable silicone resin, curable polyurethane, curable fluorosilicone, curable modified polyurethane silicones, curable modified silicone polyurethanes, curable acrylics, polytetrafluoroethylene, and the like.

Detailed Description Paragraph Right (102):

One particular type of silicone impregnant composition which is believed to be well suited for use in the impregnation step of the method of the invention is taught in U.S. Pat. Nos. 4,472,470 and 4,500,584 and in U.S. Pat. No. 4,666,765. The contents of these patents are incorporated herein by reference. Such a composition comprises in combination:

Detailed Description Paragraph Right (116):

Other suitable silicone polymer compositions are disclosed in the following U.S. Pat. Nos.:

Detailed Description Paragraph Right (118):

U.S. Pat. No. 4,108,825 discloses a composition comprising a triorganosiloxy end-blocked polydiorganosiloxane, an organohydrogensiloxane having an average of at least 2.1 silicon-bonded hydrogen atoms per molecule, a reinforcing silica filler having a surface treated with an organosilicone compound, a platinum catalyst, and ceric hydrate. Such silicone polymer composition is desirable when a web is being prepared which has flame retardant properties.

Detailed Description Paragraph Right (119):

U.S. Pat. No. 4,162,243 discloses a silicone composition of 100 parts by weight triorganosiloxy endblocked polydimethylsiloxane, reinforcing amorphous silica that is surface treated with organosiloxane groups, organohydrogensiloxane, and platinum catalyst.

Detailed Description Paragraph Right (120):

U.S. Pat. No. 4,250,075 discloses a liquid silicone polymer composition that comprises vinyl diorganosiloxy endblocked polydiorganosiloxane, polyorganohydrogensiloxane, platinum catalyst, platinum catalyst inhibitor, and carbonaceous particles. Such a silicone polymer composition is useful when a web of this invention is being prepared that has electrically conductive properties.

Detailed Description Paragraph Right (122):

U.S. Pat. No. 4,500,659 discloses a silicone composition of liquid triorganosiloxy endblocked polydimethylsiloxane wherein the triorganosiloxy units are dimethylvinylsiloxy or methylphenylvinylsiloxy, a reinforcing filler whose surface has been treated with a liquid hydroxyl end-blocked polyorganosiloxane which is fluorine-substituted, a liquid methylhydrogensiloxane, and a platinum-containing catalyst.

Detailed Description Paragraph Right (123):

U.S. Pat. No. 4,585,830 discloses an organosiloxane composition of a triorganosiloxy-endblocked polydiorganosiloxane containing at least two vinyl radicals per molecule, an organohydrogensiloxane containing at least two silicone-bonded hydrogen atoms per molecule, a platinum-containing hydrosilation catalyst, optionally a catalyst inhibitor, a finely divided silica filler, and a silica treating agent which is at least partially immiscible with said polydiorganosiloxane.

Detailed Description Paragraph Right (125):

U.S. Pat. No. 4,785,047 discloses silicone elastomers having a mixture of a liquid polydiorganosiloxane containing at least two vinyl or other ethylenically unsaturated radicals per molecule and a finely divided silica filler treated with a hexaorganodisilazane which mixture is then compounded with additional hexaorganodisiloxane.

Detailed Description Paragraph Right (126):

U.S. Pat. No. 4,329,274 discloses viscous liquid silicone polymer compositions that are believed to be suitable and which are comprised of vinyl containing diorganopolysiloxane (corresponding to component B), silicon hydride siloxane (corresponding to component A) and an effective amount of a catalyst which is a halogenated tetrameric platinum complex.

Detailed Description Paragraph Right (128):

Silicone resin compositions shown in Table I below have all been used in the practice of this invention. Such compositions of Table I are believed to involve formulations that are of the type hereinabove characterized.

Detailed Description Paragraph Right (129):

When a polymer composition of a silicone polymer and a benzophenone is impregnated into a porous web as taught herein, protection of an organic web against ultraviolet radiation is improved, and the degradation effects associated with ultraviolet light exposure are inhibited, as may be expected from prior art teachings concerning the behavior of benzophenones.

Detailed Description Paragraph Right (130):

Surprisingly and unexpectedly, however, when silicone polymer compositions such as used in this invention contain a benzophenone, the resulting composition is believed to display improved viscosity characteristics, particularly thixotropic characteristics, and also curing acceleration, when such a composition is subjected to high shear forces.

Detailed Description Paragraph Right (133):

The regulation of internal and external rheology, and of viscosity, achieved in a characteristically highly viscous polymer composition of the invention is believed to be an important and desirable feature of the benzophenone and silicone polymer compositions which find use in internally coated web manufacture as taught herein.

Detailed Description Paragraph Right (135):

Preferably, the curing proceeds to a point where the silicone polymer composition is no longer sticky, or tacky, but preferably curing is not allowed to continue to a point where the resulting polymer composition becomes excessively hard, rigid, or brittle. Compositions of this invention are controllably curable into polymeric materials which are preferably not sticky or tacky, and which have desirable elastomeric, flexural, and resiliency characteristics.

Detailed Description Paragraph Right (136):

To prepare a silicone polymer composition which incorporates a benzophenone, one preferably admixes the benzophenone with the silicone polymer composition at the time of use. The benzophenone component can be regarded as, or identified herein for convenience as, component (vi). On the same parts by weight basis above used, a composition of this invention preferably contains from about 0.3 to about 10 parts of such component (vi), although larger and smaller amounts can be used, if desired, without departing from the spirit and scope of the invention.

Detailed Description Paragraph Right (145):

The contact angle exhibited by a silicone impregnant composition of this invention varies with the particular web which is to be saturated therewith. However, the contact angle of water is generally lower for the non-impregnated side than the impregnated side. e combination of the processed web, the silicone polymer and

the fluorochemical generally produces higher water contact angles than webs treated only with fluorochemicals. The performance of an impregnant composition may be determined by the nature of a previously applied saturant such as a fluorochemical. Suitable starting compositions include 100% liquid curable silicone rubber compositions, such as SLE5600 A/B from General Electric, Mobay LSR 2580A/B, Dow Corning Silastic.sup..RTM. 595 LSR and Silastic.sup..RTM. 590 which when formulated with substituted benzophenone as taught herein will form a contact angle of much greater than 70 degrees, and typically of 90+ degrees, with typical porous webs (such as fabrics) that have a residue of fluorochemical upon (and within) the web from a prior saturation.

Detailed Description Paragraph Right (146):

The silicone polymer composition of this invention can also carry additives into the three-dimensional structure of the web during the pressured impregnation. Further, it is preferable, that any additives be bound into the cured composition permanently as located in the three-dimensional structure of the web. Particularly in the case of fabrics, this desirably positions the additives mainly on surface portions of the treated yarns and fibers in positions where they typically are beneficially located and maintained.

Detailed Description Paragraph Right (147):

Control of the pressurized impregnation step can be provided at a number of areas since the impregnation is sensitive to the viscosity of the impregnant both at atmospheric pressure and at superatmospheric pressure. The ambient temperature affecting the impregnant as it is applied, and the pressure-induced temperature changes occurring during application of the impregnant also play roles in viscosity and therefore the shear process. Of course, the chemical composition of the silicone polymer impregnant composition of this invention also plays a role in the shear process and assists in the formation of an internal coating.

Detailed Description Paragraph Right (148):

The amount of silicone polymer impregnant utilized and the weight add-on thereof are again variable and dependent upon several things such as the treated web, the desired end use of the web, cost and the like. Web weight add-ons can be as little as about 5 weight percent up to about 200 weight percent of the untreated web. For producing breathable, water-repellant fabric webs of this invention, weight add-ons are preferably in the range of about 10 to about 100 weight percent of the weight of the untreated web.

Detailed Description Paragraph Right (149):

The fluorochemical saturant composition may also contain a bonding agent. The bonding agent can facilitate the bonding of the water repellent chemical and/or the impregnate to the three-dimensional structure of the web within which it is saturated. Mobay Silopren.sup..RTM. bonding agent type LSR Z 3042 and Norsil.sup..TM. 815 primer are representative compositions that can be used to facilitate bonding of the water repellent chemicals and/or impregnant to and within the web. Use of the bonding agents is not essential to the practice of this invention, but may improve bonding of the fluorochemical and/or the silicone polymer composition to fibers.

Detailed Description Paragraph Right (150):

The fluorochemical particularly, and also the bonding agents when used, are preferably affixed to the three-dimensional structure of the web prior to a subsequent pressured impregnation. Complete affixing is not necessary for the fluorochemical. The fluorochemical will apparently facilitate the pressured impregnation of a silicone polymer composition even if the fluorochemical is not preliminarily fixed within or located within the web being treated. However, fixing, especially by sintering, appears to cause the water repellent chemicals to flow and to become better attached to the three-dimensional structure of the web. In this regard, a lesser amount of fluorochemical will remain in place better, and will better facilitate the subsequent pressure impregnation of the silicone polymer, if the sintering or insolubilizing step is performed prior to such a pressured impregnation.

Detailed Description Paragraph Right (151):

After fluorochemical saturation followed by silicone polymer impregnation and curing, a web may have a surface contact angle of greater than about 70 degrees, and more typically greater than about 90 degrees. Web impregnation pressures can involve transverse force or pressure in the range of tens to hundreds of pounds per square inch of web surface.

Detailed Description Paragraph Right (152):

Similar to the functional qualifications achieved by the use of a fluorochemical in the preferred saturating pretreatment step,, the silicone impregnant introduced by the pressured impregnation step can be defined by its functional qualifications. For example, the silicone impregnant produces a contact angle with a fluorochemical treated web of greater than about 70 degrees. In measuring the liquid contact angle with a fluorochemical treated



surface and a silicone treated surface, it will be understood that such a contact angle cannot exceed 180 degrees. The contact angle of a fluorochemical will be within a range of about 90 degrees to about 180 degrees while the contact angle of the silicone polymer will be within a range of about 70 degrees to about 180 degrees.

Detailed Description Paragraph Right (153):

The contact angle exhibited by the silicone impregnant can be, if desired, qualified against the particular web saturated with the particular fluorochemical saturant. The selection of a suitable silicone polymer composition may be determined by the nature of the previously applied fluorochemical saturant. The fluorochemical saturant and silicone polymer compositions are, however, not critical to the practice of this invention since wide respective compositional ranges may be involved. In particular, a substantially undiluted liquid silicon rubber which is available from suppliers, such as GE, Dow Corning, and Mobay-Bayer, will characteristically form a contact angle of much greater than about 70 degrees, and typically greater than about 90 degrees, with typical porous webs (such as fabrics) that have a residue of fluorochemical upon (and within) the web resulting from a prior saturation.

Detailed Description Paragraph Right (154):

The silicone polymer composition can carry additives into the three-dimensional structure of the web in the pressured impregnation step of the method of the invention. Further, the silicone polymer composition, when cured, is capable of adhering to structural elements, fibers, yarns, and the like, and any additives dispersed therein. Thus, additives are positioned adjacent to or on surfaces of structural elements, yarns, fibers and the like, in a position where they can be beneficial.

Detailed Description Paragraph Right (155):

Examples of additives that are dispersible in effective amounts in a viscous silicone polymer composition typically at a concentration of about 0.1 to 20 weight percent (based on total composition weight) include ultraviolet absorbers, flame retardants, aluminum hydroxide, filling agents, blood repellents, flattening agents, optical reflective agents, hand altering agents, biocompatible proteins, hydrolyzed silk, and the like. Hydrolyzed silk is a texturing agent that imparts a substantially silky feel to a fabric treated in accordance with the method of the invention regardless of whether or not such treated web or fabric is itself silk.

Detailed Description Paragraph Right (156):

Examples of other silicone polymer dispersible agents include those affecting thermal conductivity, radiation reflectivity, electrical conductivity, and other properties. For example, if a metallic sheen and/or thermal or electrical conductivity or infrared background blending is desired, powdered metals may be dispersed therein.

Detailed Description Paragraph Right (157):

The impregnation is sensitive to the viscosity of the silicone polymer composition. The impregnation temperature affects the silicone polymer composition by reducing or altering its viscosity. Shear-induced temperature changes occurring during application or during subsequent shear processing of the silicone polymer can affect viscosity. The chemical composition of the silicone polymer also plays a role in the treating process and can assist in the treatment of web structural elements (including fibers) and the regulation of the filling of interstices and open cell voids.

Detailed Description Paragraph Right (159):

An embodiment of a machine suitable for accomplishing pressured impregnation in accordance with this invention is illustrated in side elevational view in FIG. 4a. Two blades 200 and 210 in opposed relationship to one another are provided in functional combination with means for providing a precisely adjustable gap therebetween through which a web or fabric 300 is drawn while having a silicone polymer composition 220 applied to either one or both surfaces thereof. An enlarged side view of a typical blade 200 or 210 is shown in FIG. 4b. Dimensions A, B, C, D, and E are typically and exemplarily illustrated as, respectively, about 31/2 inches, about 11/2 inches, about 2 inches, about 1/2 inch, and about 5/16 inch. The narrow edge is preferably milled to a tolerance of about 1/10,000 inch continuously along the edge surface of each blade which is typically and illustratively about 38 inches long. Each of the corners of the narrow edge is preferably and illustratively a hard (not beveled or ground) angular edge. Each blade 200 or 210 is typically and illustratively made from carbon steel or stainless steel. A reservoir of silicone polymer composition is formed preferably on one upper surface of the fabric 300 behind (relative to the direction of fabric movement) an upper one of the blades 200 and 210 which are mounted on a frame (not shown) so as to extend horizontally. As the fabric 300 is drawn through the slit orifice defined between blades 200 and 210, some impregnant becomes entrained on the web or fabric surface and moves through such slit orifice, thereby accomplishing pressurized impregnation of web or fabric 300. The slit orifice gap is chosen preferably and illustratively to be slightly smaller than the relaxed



thickness of the starting web or fabric.

Detailed Description Paragraph Right (160):

Referring to FIG. 4a, a second pressured impregnation station is seen to be positioned downstream (relative to the direction of fabric movement) from the pair of opposed blades 200 and 210. At this station, a knife blade 230 is provided which has an edge that presses against the web or fabric 300 to reintroduce the silicone polymer composition into the fabric 300. One side of blade 230 adjacent the edge thereof is strongly biased against an adjacent cylinder or bar 240, which, in the embodiment shown, does not rotate. If desired, bar 240 can be journaled for rotational movement. As the fabric is moved between the blade 230 and the bar 240, it is preferably uniformly compressed. Preferably, the compression force is in the range of about 10 to about 500 inch pounds, although higher and lower forces can be employed. As the fabric 300 passes over the edge of blade 230, it is drawn away at an angle from the blade edge under longitudinal tension. For example, longitudinal tension in the range of from about 0.5 to 10 pounds per inch can be employed. Such pressured impregnation serves to distribute and reintroduce the polymer composition in the web. Excess polymer composition is removed by blade scraping. Passage of the fabric 300 between the blade 230 and the bar 240 and over the edge of the blade 230 is believed to produce shear forces in the impregnant 220 (within the fabric 300) that facilitate flow and distribution thereof within the three-dimensional matrix of the fabric 300. Concurrently, blade 230 also scrapes excess silicone polymer composition impregnant off the fabric's surface in contact with the edge of blade 230.

Detailed Description Paragraph Right (161):

Both the steps of fluorochemical saturation and of subsequent silicone polymer composition impregnation are performable, if desired, in production volumes, and at speeds which can be typical of the so called high end range of fabric finishing lines. The fluorochemical saturation is conveniently accomplished conventionally by using a padbath in which the fabric is run through a dilute treating bath followed by squeeze rollers to remove excess liquid and overdrying. In general, any method of applying the fluorochemical would be acceptable.

Detailed Description Paragraph Right (162):

Another embodiment of a machine suitable for accomplishing pressurized impregnation in accordance with this invention is shown diagrammatically in FIG. 5 which also illustrates a process embodiment of this invention. At an impregnation head, pressurized introduction of the silicone polymer composition into the web is first carried out. At a subsequent stage, controlled pressure reintroduction, distribution, and metering of the silicone polymer impregnant and recovery of excess impregnant transpires using a shear knife or blade which applies transverse force against the impregnated web laterally across the web. In a subsequent stage, further controlled pressure reintroduction and metering takes place by means of a flexible blade, such as a so-called flex-knife or Spanish knife. Here, additional recovery of excess liquid impregnant is accomplished. In all knife-applying states, the excess impregnant removed is collected and preferably passed by a recycling system back to the initial, pressured introduction stage to achieve process operating economies. Still further successive impregnant pressure reintroduction stages may be used if desired. The direction of the arrows in the diagrammatic representation of FIG. 5 shows the general direction of movements in the region of the impregnation head, including the general direction of impregnant movement in the practice of such process.

Detailed Description Paragraph Right (163):

The apparatus employed in the present invention functions first to apply and preferably concurrently impregnate a silicone polymer composition into a web under pressure. Such silicone polymer composition is then reintroduced, distributed, and metered in a controlled manner in the web with the aid of transversely applied shearing force and compressive force such that the impregnated composition becomes distributed in the web so that an internal layer of silicone is formed while the fibers are at least partially enveloped while the interstices or open cells are substantially completely filled with the silicone polymer composition in the region of the internal coating. During treatment, the web is longitudinally tensioned and the pressurized application and impregnation and the subsequent shearing and compressive actions are successively accomplished in localized zones preferably extending generally laterally across the web (that is, generally perpendicularly to the direction of such longitudinal web tensioning) using transversely applied force exerted locally against surface portions of the web during each impregnation and shearing operation. The web is conveniently and preferably, but not necessarily, moved longitudinally relative to such laterally extending web processing zones. The impregnation, shearing and compressing steps are preferably carried out successively or sequentially. Such zones are themselves preferably at stationary locations while the web is moved, but if desired, the web can be stationary while the zones are moved, or both. The result is that the silicone polymer composition impregnant flows into the web and is distributed internally generally uniformly to a predeterminable and controllable extent.

Detailed Description Paragraph Right (164):

A schematic side elevational view of another embodiment of a suitable pressurized impregnation machine for use in the practice of the invention is shown in FIG. 6. This machine continuously moves a longitudinally tensioned web 60 successively through a pressure impregnation station which incorporates a reverse roll coater having rollers 10 and 11, a shear station which incorporates a shear knife 20, and a finishing station which employs at least one so called flex-knife (or Spanish knife) 30. Optionally, but preferably (for reasons of process operating economics) excess silicone polymer composition that is removed from web surfaces in the shear station and finishing station is returned to the pressure impregnation station for reuse using liquid recovery and recycle system 40. In the pressure impregnation station, a silicone resin impregnant 50 is contained within reservoir 51. Roller 12 rotates in the indicated direction so that its circumferential surface, preferably a textured or gravure surface, picks up liquid 50 from reservoir 51 and deposits it on the circumferential surface of roller 10 across a controlled width gap 13 between rollers 10 and 12. Typically, gap 13 is actually less than the unencumbered thickness of the starting web 60. Roller 10 also preferably has a textured or gravure surface. Roller 10, rotating in the roller arrow indicated direction, which is opposite to the direction of travel of web 60, applies the silicone polymer impregnant to one surface of the moving web 60, which is typically a fabric. Roller 11 is urged with a compressive force against the back or opposed surface of web 60 and roller 11 rotates in a direction which is the same as that in which web 60 travels. Roller 11 aids in achieving the desired pressured impregnation of web 60 by impregnant from the surface of roller 10.

Detailed Description Paragraph Right (165):

Referring to FIG. 6, the impregnant is believed to be introduced into the web and into the interstices or open cells of the web 60 by the aid of a back-pulling or shearing action resulting from the distorting and pressuring of web 60 caused by rollers 10 and 11 rotating in the same direction. This direction may be the indicated direction with roller 10 rotating against the linear movement of web 60 indicated by web directional arrow 61, or all rollers 10, 11 and 12 may be reversed in respective rotational direction so as to cause each roll to turn in an opposite direction relative to that direction which is illustrated by the respective roller arrows in FIG. 6. Regardless of which side of web 60 is back-pulled or subjected to shearing action by a reverse rotating roller, the web 60 is stretched and distorted to pull open the interstices of the web and to aid in impregnating web 60 with silicone polymer impregnant 50. This distorting, and particularly this stretching, is believed to facilitate the full and deep introduction of the impregnating liquid into the moving web 60.

Detailed Description Paragraph Right (166):

The extent of pressured impregnation of the silicone polymer impregnant 50 into the web 60 which occurs between rotating rollers 10 and 11 is controllable to some extent by such variables as the speed of roller rotation, the pressure exerted by rollers 10 and 11 on web 60, the durometer hardness and surface characteristics of each roll 10 and 11 (particularly of the preferred textured or gravure surface of roll 10). However, the pressurized impregnation may also be carried out with rollers 10 and 11 which have finely milled, smooth circumferential surfaces. The viscosity of impregnant liquid 50 and the amount of impregnant liquid 50 transferred from roll 12 to roll 10 across gap 13 may also be varied to regulate impregnation. Feed roller 12 preferably rotates counter to application roller 10. The impregnant 50 can be monitored to assure that its homogeneous composition is maintained. If desired, the impregnant 50 formulation can be altered to adjust to process needs during a continuous treating operation.

Detailed Description Paragraph Right (167):

The result of the pressured web 60 impregnation which is accomplished between rollers 10 and 11 using a silicone polymer composition impregnant 50, which can have the viscosity or consistency of a conventional bathtub caulk composition, is to produce a web 60, or fabric, whose interstices or open cells are substantially completely filled with impregnant in the region of the internal coating. In, for example, the case of a fabric, the impregnation extent can be such that spaces (i.e., interstices or open cells) between the fabric's fibers/filaments, or the fabric's yarn members (as the case may be) are filled with impregnant 50. However, the amount of impregnant 50 which is thus introduced into web 60 can be much less than a saturation level; for example, the amount introduced can be insufficient even to coat or substantially completely envelope individual fibers of the web. Actually, the impregnant 50 can be relatively non-uniformly distributed in the web after such pressurized impregnation. The action of the shear knife 20 in the next zone of processing is such as to smooth out and to make uniform the distribution of impregnant 50 in web 60. Also, the shear knife 20 helps regulate the amount of impregnant 50 that is allowed to remain in web 60.

Detailed Description Paragraph Right (168):

After the shear zone, if desired, a top coat polymer can additionally be introduced; for example, just before or after a flex knife 30. By overcoating, for example, the original impregnant with a dilute or very thin second or top

coat, a more tightly cross linked impregnated or enveloped product may be achieved, or surface properties of the product can be varied or improved. For example, the top coating can comprise a dilute dispersion of a fluorochemical fabric treating composition. In a web treated therewith, such treatment enhances surface properties of the web, such as by increasing grease or chemical penetration resistance, or soil resistance, or the like. The dilute fluorochemical dispersion can be applied by spraying, misting, or the like. Both treating agents then enter a curing stage, which can be accomplished conveniently by passing the treated web through an oven wherein the temperature and web residence time are sufficient to cure both the fluorochemical and silicone polymer impregnants to a desired extent, or by radiation, if desired.

Detailed Description Paragraph Right (169):

The amount of silicone polymer impregnant actually introduced through the pressured impregnation, and into the preferably stretched openings of the interstices of the web 60 is influenced by such factors as the velocity of movement of web 60, the viscosity characteristics of impregnant 50, the compressive pressure exerted by roll 10 against roll 11, the longitudinal tension exerted upon the tensioned web 60, impregnant distribution achieved by shear blade 20 and by scraper flex knife(s) 30, and the like. In particular, the impregnant reintroduction and distribution believed to be achieved by bar or shear knife 20 is achieved by the exertion of a pressure against moving tensioned web 60. The shear force and the temperature elevation due to such shear force results in the impregnant 50 flowing upon the three dimensional structure of the web 60 and the knife 20.

Detailed Description Paragraph Right (170):

Preferably, the impregnant 50 is thixotropic. The flowing of the impregnant 50 into the web 60 using controlled liquid rheology preferably does not result at the time of impregnation in a fluid viscosity which is so low as to cause the impregnant to spread into and be distributed substantially uncontrolled throughout the web 60. However, the flowing activity of the impregnant is preferably accomplished using an impregnant 50 which has a controllable rheology and viscosity such that an impregnant 50 will achieve a desired envelopment of individual fibers of the web 60. Particularly when the web 60 is a fabric, this envelopment is preferably a surrounding of the fabric's individual fibers with a localized layer or film of silicone polymer while an internal layer is formed.

Detailed Description Paragraph Right (173):

Another embodiment of a machine suitable for use in the practice of this invention is shown schematically in side elevation in FIG. 7. In this embodiment, rollers 10 and 11 of the FIG. 6 apparatus are replaced with a combination of a reservoir 51, and a bar or shear knife 100. The reintroduction bar or shear knife 100 pressurizes the impregnant liquid 50 which is applied or deposited onto the moving web 60 from the reservoir 51 as a liquid or bath. The web 60 in effect constitutes a retaining wall for a part of the reservoir 51. The reservoir 51 thus functions to hold a pool of the silicone polymer composition impregnant 50 against a surface of the moving web 60 which in the embodiment shown, is moving vertically upwardly. The bar or shear knife 100 functions to apply pressure or force upon the silicone polymer composition impregnant 50 that was deposited on the web 60, thereby to cause the impregnant 50 to penetrate the web 60. The knife 100 also serves to distribute and move the impregnant in the web and to accomplish envelopment of the fibers thereof. Excess impregnant 50 is also scraped away by knife 100. Optionally, one or more of flex knives 100 function to further reintroduce, distribute the impregnant 50 and to envelope fibers of web 60 while forming an internal silicone polymer coating within the web. The knives 110 can be considered to function in a manner which is equivalent to the knives 30 on the treated surface of web 50 in the FIG. 6 apparatus.

Detailed Description Paragraph Right (175):

Relative to the FIG. 7 embodiment, the FIG. 6 embodiment is believed to exhibit a wider degree of control in the practice of the present impregnation process. Particularly, both the initial applied amount and the successive pressurings of, a silicone polymer impregnant 50 are precisely controllable. Relative to the FIG. 6 embodiment, the FIG. 7 embodiment is characterized by the capability for operation at higher web 60 transport speeds, typically at speeds characteristic of higher end commercial fabric finishing line operations. The embodiment shown in FIG. 6 is believed to be suitable for producing internally coated fabrics when the fabrics are of the thicknesses characteristic of garments, and where deeply controlled pressured impregnation over distances extending perpendicularly into and through a web of fabric greater than about 1/16 inch is not generally required.

Detailed Description Paragraph Right (178):

In a first functional processing station 78, a silicone polymer composition is applied to one face (here, the upper face 79) of web 74 by a conventional reverse roll coater apparatus 81 wherein such composition is applied to the surface of a reversely rotating (relative to the direction of travel for web 74) coating roll 82 from a nip region reservoir 83 formed between the coating roll 82 and a transfer roll 84 (which rotates in the direction of travel for

web 74, but whose surface does not contact web 74). The web 74 is transversely compressed between coating roll 82 and drive roll 86 as it passes through station 78. Thus, the polymer composition is applied under a positive pressure against face 79 by coating roll 82 which functions to cause the composition to be impregnated into web 74. A present preference is to use a coating roll 82 which has smooth, chrome plated surfaces.

Detailed Description Paragraph Right (188):

Those skilled in the art will appreciate that the amount of shear force applied by a shear knife 101 or 102 transversely against a web 74 is a function of many variables with probably the most important or principal variables being the fluorochemical pretreatment, the silicone polymer viscosity and the longitudinal web tension (assuming a fixed spatial position for idler rolls 105, 106 and 107 and shear knives 101 and 102 during operation).

Detailed Description Paragraph Right (189):

When a suitable and preferred level of applied shear force and web tensioning has been achieved to produce a product having enveloped fibers and an internal silicone coating, one can usually hear a distinctive sound in the region of a shear blade 101 and 102. This sound can also be heard in the vicinity of shear blades being used in the operation of other processes described herein. This sound can in fact be used by an operator as a rough guide as to whether or not he is succeeding in producing a silicone polymer impregnated product containing enveloped fibers and an internal coating.

Detailed Description Paragraph Right (197):

The oven 119 functions to cure the silicone polymer composition thus impregnated into web 74. Oven 119 can be operated with gas or other energy source. Oven 119 can extend for from about 12 to 20 yards, a 15 yard long oven being convenient.

Detailed Description Paragraph Right (198):

Curing temperatures of from about 320.degree. to about 500.degree. F., applied for times of from about 2 minutes to about 30 seconds (depending upon the temperature) are desirable. If a curing accelerator is present in the silicone polymer, curing temperatures can be dropped down to temperatures of about 265.degree. F. or even lower (with times remaining in the range indicated).

Detailed Description Paragraph Right (204):

In FIG. 12b, a further stage of web pressurization is introduced after the flex knife 112' and before the tenter frame 118'. Here, the web 74' after passage through the flex knife 112' is passed through the nip region 126 existing between padder roll 111' and associated transfer roll 127 where the web 74' is subjected to compression between such rolls 127 and 111' for the purpose of achieving a better distribution of silicone polymer composition on web 74'.

Detailed Description Paragraph Right (209):

A presently preferred web which is both fluorochemical and silicone resin treated and which is breathable, water resistant and rewashable is characterized as being a longitudinally tensionable porous flexible fibrous web having opposed, substantially parallel surfaces that are comprised of associated fibers with interstices between the fibers or is a matrix having cells or pores therein. The web is substantially uniformly impregnated with a fluorochemical and thereafter impregnated with a silicone polymer composition, to form a web having an internal layer within the web wherein the outer surfaces of the web are substantially full of silicone polymer and the web is breathable and water resistant or waterproof. At least a portion of the fibers or cell walls are enveloped. At least one surface thereof is characterized by having a visual appearance which is substantially the same as the visual appearance of one surface of the starting porous web.

Detailed Description Paragraph Right (212):

A general process for making a porous web of this invention comprises the steps of: tensioning a flexible, porous web as above characterized, applying a curable silicone polymer composition to at least one web surface and then moving over and against one surface of the tensioned web a uniformly applied localized shear force to: uniformly distribute the composition within the web, at least partially individually envelope surface portions of at least some of said fibers or passageways through the web matrix with said composition in a desired web internal region. Thereafter, the web is subjected to conditions sufficient to cure the composition in said web. Curing is accomplished by heat, by radiation, or both.

Detailed Description Paragraph Right (213):

A presently preferred process for making a fluorochemical and silicone resin treated web having breathability,

water resistance and rewashability which is adapted for continuous operation comprises the successive steps of: impregnating the web with a fluorochemical, longitudinally tensioning the fluorochemical impregnated web while sequentially first applying to one surface thereof a curable silicone polymer composition and concurrently applying a transversely exerted localized compressive force against said surface, and moving over said surface of the web substantially rigid shearing means which exerts transversely an applied, localized shear force against said surface and wipes away exposed portions of silicone polymer composition on said surface, thereby forming an internal layer of silicone polymer while enveloping at least some of the fibers or passageways through the matrix; and curing the silicone polymer composition in the web.

Detailed Description Paragraph Right (217):

One aspect of the invention is a recognition that when high forces are applied to curable silicone polymer compositions, such as those as viscous as bathtub caulk, then the viscosities of these materials can be lowered perhaps up to 99% or more. Conversely, when cured (polymerized and/or cross-linked) these compounds increase in viscosity up to perhaps 1,000,000% or more. The internal and external rheological control of silicone impregnant materials achieved by the present invention is believed to be of an extreme level even for thixotropes. When subjected to shear force, the liquid silicone polymer composition can flow more readily, perhaps comparably, to water. When subsequently subjected to curing, the same liquid composition sets to a solid form which can have a consistency comparable to that of a hard elastomeric rubber.

Detailed Description Paragraph Right (218):

The invention preferably employs a combination of: (i) mechanical pressure to squeeze a silicone polymer composition impregnant into a porous web; (ii) a porous web pretreatment with a water repellent chemical, such as a fluorochemical, which is theorized to reduce the surface tension characteristics of the web and create a favorable surface contact angle between the silicone polymer composition and the treated web which subsequently allows, under pressure and shear force exerted upon an applied silicone polymer composition, the production and creation of an internal coating or layer which envelopes fibers or lines cell walls in a localized region within the web as a result of impregnant flow in the web; and (iii) a liquid silicone polymer composition impregnant preferably having favorable rheological and viscosity properties which responds to such working pressures and forces, and in controllably impregnated into, and distributed in a web. This combination produces a web having the capability for a high degree of performance. This product is achieved through pressure impregnation and applied shear forces brought to bear upon a web so as to cause controlled movement and flow of a silicone polymer composition into and through a web. Preferably, repeated compressive applications of pressure or successive applications of localized shear forces upon the impregnant in the web are employed.

Detailed Description Paragraph Right (221):

Thixotropic behavior is preferably built into an impregnant used in the invention by either polymer design or additive/filler design. For example, it now appears that thixotropic behavior can be accentuated by introducing into a silicone polymer composition certain additives that are believed to impart enhanced thixotropy to the resulting composition. A lower viscosity at high shear rates (during application to a web) is believed to facilitate impregnant flow and application to a web, whereas an impregnant with high viscosity, or applied at a low shear rate (before and/or after application) actually may retard or prevent structural element (including fiber) envelopment.

Detailed Description Paragraph Right (223):

In stage 1, silicone polymer composition impregnant is prepared can be purchased commercially and comes in typically two parts, designated as A and B. For example, in a silicone polymer composition as taught in U.S. Pat. No. 4,472,470, a base vinyl terminated polysiloxane is the A part, while a liquid organohydrogensiloxane crosslinking agent is the B part. Certain remaining components, such as a resinous organopolysiloxane copolymer and a platinum catalyst may (or can) apparently initially be in either part A or part B.

Detailed Description Paragraph Right (224):

Stage 2 can be considered to involve the mixing of such a product's parts with or without additives. Changes in viscosity can be obtained and measured based on applied shear rates and shear stresses. Such changes can be experienced by an impregnant with or without additives. Up to a 99% reduction in viscosity of a liquid silicone polymer composition is believed to be obtainable by the shear forces involved in the mixing or infusion of a silicone polymer composition impregnant into a web during, the elapsed applied combination of processing time, temperature, radiation, and/or chemical changes involved. Thereafter, a very substantial increase in impregnant viscosity is believed to be obtainable taking into account these same factors. Normally, the most significant factor is now believed to be the mixing shear gradient that typically reduces the viscosity of the impregnant about 50% below the starting or rest viscosity.

Detailed Description Paragraph Right (227):

Stage 5 can be considered to be a second stage internal matrix dispersing and reintroduction with metering and also recovery and recycling of excess impregnant. The variations in the viscosity of the impregnant are equivalent to stage 4. The viscosity of the impregnant is again lowered causing it to flow within the web. Because of the application of repeated shear force induced reductions in viscosity, the thixotropic behavior of an impregnant may not undergo complete recovery, following each application of shear force and the viscosity of the impregnant may not revert to its pre-impregnation values. The silicone polymer composition impregnant is believed to have the capacity to form enveloping internal coating in a predetermined region wherein the interstices or open cells are substantially completely filled within the three-dimensional matrix constituting a web during the time intervals that the impregnant is caused to flow under pressure in and about matrix components. In between these times, the impregnant may recover substantially all of its initial high viscosity, although perhaps slightly less so with each repeated application of shearing pressure or force.

Detailed Description Paragraph Right (230):

FIG. 8, consisting of FIGS. 8a through 8d, shows four graphs illustrating four ways that could be used for plotting impregnant rheological behavior: (a) shear rate versus shear stress (uniform scales), (b) shear rate versus shear stress (log scales), (c) viscosity versus shear rate (uniform scales), and (d) viscosity versus shear rate (log scales), if desired, in the practice of this invention. Only the log versus log scales are believed to be capable of encompassing a full range of values for the three indicated variables. The graphs represent some broad ranges of viscosity changes relative to shear stress that could be undergone by a given silicone polymer composition impregnant during execution of a given pressured impregnation procedure as taught herein.

Detailed Description Paragraph Right (232):

Surface tension is believed to induce wetting effects which can influence the behavior of a silicone polymer composition impregnant relative to the formation of a fiber enveloped layer therewith in a fibrous porous web. For example, adhesion is theorized to be a wetting effect. Spontaneous adhesion always occurs for contact angles less than about 90.degree.. However, for a combination of a rough surface and a contact angle over 90.degree., adhesion may or may not occur. In fact, roughness becomes antagonistic to adhesion, and adhesion becomes less probable as roughness increases.

Detailed Description Paragraph Right (235):

FIG. 9 is a schematic vector diagram illustrating the surface tension forces acting at the vertex boundary line of a liquid contact angle on a planar solid surface. It illustrates how surface tension forces might be measured between a silicone polymer composition impregnant and a fiber of a web (or a fabric) as treated by the invention.

Detailed Description Paragraph Right (237):

Regions of adhesion versus adhesion, penetration versus repellency, and spreading versus retraction are shown by shaded areas. FIG. 10 illustrates what is theorized to be the relationship of a silicone polymer composition impregnant to silicone polymer composition solids in a treated web as regards such factors as adhesion, penetration, spreading, and retraction.

Detailed Description Paragraph Right (239):

FIGS. 11a through 11d show a broad range of illustrative flow characteristics that could be demonstrated by silicone polymer composition impregnants suitable for use in this invention using pressured impregnation of a web as taught herein.

Detailed Description Paragraph Right (244):

Liquid Silicone Polymer Preparation

Detailed Description Paragraph Right (245):

100 parts by weight of the curable liquid silicone polymer available commercially from Mobay as "Silopren.sup.RTM. LSR 2530" was mixed in a 1:1 ratio, as recommended by the manufacturer. A Hockmayer F dispersion blade at low torque and high shear was used to do the mixing. To this mixture were added 5 parts by weight of BSF "Uvinul 400" and 5/10 parts by weight Dow Corning 7127 accelerator, believed to be a polysiloxane but containing an undisclosed active accelerated ingredient.

Detailed Description Paragraph Right (246):

Liquid Silicone Polymer Preparation



Detailed Description Paragraph Right (247):

The procedure of Example 1 was repeated with various other curable viscous liquid silicone polymer composition commercially available. To this product system is added a substituted benzophenone and other additives, the result of which are shown in Table VII below. All parts are by weight.

Detailed Description Paragraph Right (255):

Next, the fabric was heated in an oven for 1 minute at 350.degree. F. This heating sinters the water repellant fluorochemical. The fabric with its fluorochemical residue is then run as in the preferred production embodiment, FIG. 7, in a vertical configuration and is described below. The fabric is run from a roll that incorporates significant braking or clutching to initiate the tension required for controlled material alignment and coating during application. The fabric web travels through a series of idler rolls ending at the application trough. As it passes the application trough, it picks up a thin coating of silicone impregnant and then moves under a shear blade that is parallel to the floor. The silicone impregnant is applied at 1.0 oz/sq. yd. and continues under a flex blade that is also parallel to the floor.

Detailed Description Paragraph Right (257):

Finally, the impregnated fabric was run through a line oven, of approximately 10 yards in length, at 4-6 yards per minute, and was cured at 325.degree.-350.degree. F. It then passes through a series of idler rollers and is rolled up on a take-up roll, completing the tension zone. The resultant fabric has a non-tacky thin film of silicone that was internally coated to form a fiber encapsulated, interstice-filled fabric.

Detailed Description Paragraph Right (262):

FIG. 3a is a photomicrograph of the gold color Tactel fabric described in Example 20. The surface of the material as been magnified 120 times and shows that the cured silicone polymer impregnant is present as a thin film, or coating, or layer within the material and envelopes at least a portion of the fibers. The fiber bundles are somewhat distinguishable in the weave, but each filament in the fiber bundles is not individually distinct.

Detailed Description Paragraph Right (263):

The sample in FIG. 3b has been magnified 600 times and shows the cross-section of a fiber bundle from the same Gold Tactel in FIG. 3a. The cured silicone polymer impregnant envelopes at least a portion of the fibers. The interstices or void areas between filaments in the region of the internal coating are mostly filled or plugged by such impregnant. However, the web remains breathable and because of the impregnant barrier is either water resistant or waterproof.

Detailed Description Paragraph Right (264):

FIG. 3c is the side of the fabric in FIG. 1 opposite from which the silicone polymer impregnant was applied. The silicone polymer impregnant is most readily apparent at the fiber bundle interstices and not visible in the fiber bundles themselves.

Detailed Description Paragraph Right (274):

Prior Art Silicone Polymer Treated Fabric

Detailed Description Paragraph Right (275):

The fabric resulting from a prior art application of a viscous liquid curable silicone polymer composition is shown in FIG. 2. The photographic view of FIG. 2 is at 150X magnification. It shows a polyester and cotton cloth blend into which Dow Corning 590 LSR silicone polymer composition has been coated by a procedure of the prior art. The fabric side shown in FIG. 2 is the top, or treatment, side, which was the fabric side upon which coating was accomplished.

Detailed Description Paragraph Right (276):

As shown by the example of the treated fabric of FIG. 2, the prior art impregnated fabric is characterized by a high degree of disorder. A large number of particulates (typical) appear to litter the surface of the fabric. A substantial portion of the area of the surface, which appears to be a solid layer, is silicone polymer composition. Certain yarn fragments can be observed to protrude through the surface of this silicone polymer composition. Additionally, the silicone polymer composition on either the polyester or the cotton fibers is not an encapsulation layer, but rather a matrix with the coated fibers being in general disarray, probably from forces occurring during the indicated prior art silicone polymer composition application procedure. Although silicone polymer composition is present upon the yarn or fiber surfaces of the substrate, and certainly is present as a layer upon the exterior surface of the three-dimensional fabric body, the silicone polymer composition has not controllably

and individually encapsulated the fibers and left the interstices between fibers largely devoid of such polymer. In the prior art, a placement of silicone polymer composition in a fabric is not controlled to such a degree so as to produce a product in accordance with the present invention.

Detailed Description Paragraph Right (279):

FIG. 13a is a photomicrograph of the Tactel (hot coral) fabric described in Example 23. The surface of the material has been magnified 120 times and shows that the cured silicone polymer impregnant is present as a thin film, or coating, or layer within the material and envelopes at least a portion of the fibers. The fiber bundles are somewhat distinguishable in the weave, but each filament in the fiber bundles is not individually distinct.

Detailed Description Paragraph Right (280):

The sample in FIG. 13b has been magnified 800 times and shows the cross-section of a fiber bundle from the same Tactel in FIG. 13a. The cured silicone polymer impregnant envelopes at least a portion of the fibers. The interstices or void areas between filaments in the region of the internal coating are mostly filled or plugged by such impregnant. However, the web remains breathable and because of the impregnant barrier is either water resistant or waterproof.

Detailed Description Paragraph Right (281):

FIG. 13c is the side of the fabric in FIG. 1 opposite from which the silicone polymer impregnant was applied. The silicone polymer impregnant is most readily apparent at the fiber bundle interstices and not visible in the fiber bundles themselves.

Detailed Description Paragraph Right (295):

The results of abrasion resisting testing clearly show that enveloped fiber fabrics of this invention have superior wear characteristics compared to the untreated original (starting) fabrics. In most cases, the enveloped fiber fabric samples underwent twice as many cycles as the untreated samples without evidencing tearing in the samples. Such results can be explained by theorizing that the envelope with silicone polymer of the yarns and fibers comprising a fabric, provides such treated yarns and fibers with a lubricity agent so that abrasive action was minimized and the integrity of the fabric was preserved significantly longer. The anti-abrasion character also applied to the minimized effects of one fiber rubbing against another fiber, or of one yarn against another yarn.

Detailed Description Paragraph Center (14):

drastically. Excellent "hand" was retained after the test. It is believed that pretreatment with a fluorochemical having good water repellent properties can augment and even synergistically coact with the silicone resin used to produce fiber enveloped fabrics of this invention to produce superior spray ratings in such a fiber. The results are shown in the following Table.

Detailed Description Paragraph Table (1):

TABLE I \_\_\_\_\_ Illustrative Starting Silicone Polymer Compositions  
Trade Manufacturer Designation Components.sup.(1) \_\_\_\_\_ Mobay  
Silopren .RTM. Vinyl-terminated LSR 2530 polydimethyl/siloxane with fumed silica, methylhydrogen polysiloxane  
Mobay Silopren .RTM. LSR 2540/01 Dow Corning Silastic .RTM. polysiloxane 595 LSR General SLE 5100  
polysiloxane Electric General SLE 5106 siloxane resin Electric solution General SLE 5300 polysiloxane Electric  
General SLE 5500 polysiloxane Electric Shin-Etsu KE 1917 Shin-Etsu DI 1940-30 SWS Liquid Rubber silicone  
fluid with Silicones BC-10 silicone dioxide Corporation filler and curing agents

\_\_\_\_\_ Table I footnote: .sup.(1) Identified components do not  
represent complete composition of the individual products shown.

Detailed Description Paragraph Table (4):

TABLE IV \_\_\_\_\_ MACHINE ELEMENT PARAMETERS Second Stage  
Reintroduction Bar Knife: Adjustment Variability Effect of Parameters to Adjustment Adjustment of Web  
\_\_\_\_\_ Angle of bar knife Bar knife faces Angle effects shear forward  
to meet forces on impregnate web coming to and determines bar; bar knife distortion or vertical to web, bar  
agitation as it faces away or relates to causing racked back from flow of impregnate; web run direc- can  
determine tion reintroduction of impregnate by dammed quantity of impregnate Edge Shape Knife very  
Sharpness of knife sharp affects shear forces. The sharper and thinner the edge, the greater the shear forces at  
the contact edge Pressure Relates to web Greater pressure tautness increases forces at contact edge

Detailed Description Paragraph Table (6):



TABLE VI

Illustrative Silicone Resin Compositions Mixture Ratio Substituted Other Example Starting of Packaged Benzophenone Additives # Silicone Resin Components.sup.(1) Name Parts Name Parts

1 Silopren .RTM.  
LSR 2530 1:1 Uvinul 400 5 7127 Accelerator 5/10 2 Silastic .RTM.595 LSR 1:1 Uvinul 400 5 Syl-off .RTM. 7611.sup.(2) 50 3 SLE 5100 10:1 Uvinul 400 5 Sylox .RTM. 2.sup.(3) 8 Liquid BC-10 1:1.sup.1:1 4 Silopren .RTM. LSR 2530 1:1 Uvinul 400 5 Hydral .RTM. 710.sup.(4) 10 5 Silopren .RTM. LSR 1530 1:1 Uvinul 400 5 Silopren .RTM. LSR 1 Z3042.sup.(5) 6 SLE 5500 10:1 Uvinul 400 5 7 Silopren .RTM. 2540 1:1 Uvinul 400 5 8 SLE 5300 10:1 Uvinul 400 5 9 SLE 5106 10:1 Uvinul 400 5 10 Silopren .RTM. LSR 2530 1:1 Uvinul 400 5 Flattening Agent 4 OK412 .RTM..sup.(6) 11 Silopren .RTM. LSR 2530 1:1 Uvinul 400 5 Nalco.sup.(5) 1SJ-612 50 Colloidal Silica.sup.(7) 12 Silopren .RTM. LSR 2530 1:1 Uvinul 400 5 Nalco .RTM. 1SJ-614 Colloidal Alumina.sup.(8) 13 Silastic .RTM. 595 LSR 1:1 Uvinul 400 5 200 Fluid.sup.(7) 7 14 Silopren .RTM.LSR 2530 1:1 Uvinul 400 5 15 Silastic .RTM. 595 LSR 1:1 Uvinul 400 5 Zepel .RTM. 7040.sup.(10) 3 16 Silastic .RTM. 595 LSR 1:1 Uvinul 400 5 Zonyl .RTM. UR.sup.(11) 1/10 17 Silastic .RTM. 595 LSR 1:1 Uvinul 400 5 Zonyl .RTM. FSN-100.sup.(12) 1/10 18 Silopren .RTM. LSR 2530 1:1 Uvinul 400 5 DLX-600 .RTM..sup.(13) 5 19 Silopren .RTM. LSR 2530 1:1 Uvinul 400 5 TE-3608 .RTM..sup.(14) 5

Table VI

Footnotes: .sup.(1) Ratio listed is that recommended by the manufacturer. .sup.(2) Syloff .RTM. (registered trademark of Dow Corning) is a crosslinker. .sup.(3) Sylox .RTM. 2 (registered trademark of W.R. Grace & Co.) is a synthetic amorphous silica. .sup.(4) Hydral .RTM. 710 (registered trademark of Alcoa) is a hydrated aluminum oxide. .sup.(5) Silopren .RTM. LSR Z/3042 (registered trademark of Mobay) is a silicone primer (bonding agent) mixture. .sup.(6) Flattening Agent OK412 .RTM. (registered trademark of Degussa Corp.) is a wax coated silicon dioxide. .sup.(7) Nalco .RTM. 1SJ612 Colloidal Silica (registered trademark of Nalco Chemical Company) is an aqueous solution of silica and alumina. .sup.(8) Nalco .RTM. 1SJ614 Colloidal Alumina (registered trademark of Nalco Chemical Company) is an aqueous colloidal alumina dispersion. .sup.(9) 200 Fluid (registered trademark of Dow Corning) is a 100 centistoke viscosity dimethylpolysiloxane. .sup.(10) Zepel .RTM. 7040 (registered trademark of duPont) is a nonionic fluoropolymer. .sup.(11) Zonyl .RTM. UR (registered trademark of duPont) is an anionic fluorosurfactant. .sup.(12) Zonyl .RTM. FSN100 (registered trademark of duPont) is a nonionic fluorosurfactant. .sup.(13) DLX6000 .RTM. (registered trademark of duPont) is a polytetrafluoroethylene micropowder. .sup.(14) TE3608 .RTM. (registered trademark of duPont) is a polytetrafluoroethylene micropowder.

#### Other Reference Publication (2):

Uvinul UV Inhibitors for Cosmetics, Plastics, Coatings, Textiles; BASF Corporation, Rensselaer, NY.

#### CLAIMS:

1. A flexible porous web which has an internally located coating that is comprised of a curable silicone polymer composition, said coating being positioned in an approximately planar region extending through the web in a direction generally parallel to and spaced from at least one major surface thereof, said web, upon curing the said curable composition thereof, being breathable and highly water repellent and exhibiting a hand and flexibility comparable to the hand flexibility of an untreated web.

2. The web of claim 1 wherein said liquid silicone polymer composition has a viscosity of at lesat about 5,000 and less than about 2,000,000 centipoise and the quantity of said silicone polymer composition ranges from about 5 to about 200 weight percent of the weight of the untreated web.

3. The web produced by curing the curable polymer-containing web of claim 2 wherein said silicone polymer composition is cured and is elastomeric.

24. A process for producing a porous web that is internally coated with a silicone polymer composition comprising the steps of continuously and successively;

(a) tensioning a porous web;

(b) uniformly applying, under pressure, to one surface of said tensioned web a curable silicone polymer composition; and

(c) applying a transverse shear force uniformly and laterally across and against said surface and concurrently wiping away excess portions of silicone polymer from said surface;

where, an internal layer of silicone polymer is formed in an approximately planar region extending through said web in a direction generally parallel to space from at least one major surface thereof.

25. The process of claim 22 wherein, a compressive force is applied uniformly and laterally across and against one surface of said web that wipes away excess portions of said liquid on said surface.

26. The process of claim 24 wherein the amount of said silicone polymer composition in the product web in the range of about 5 to about 200 weight percent of the weight of the untreated web.

27. The process of claim 24 comprising an additional step wherein the silicone polymer composition is cured in said web.

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L8: Entry 7 of 7

File: USPT

Oct 19, 1982

DOCUMENT-IDENTIFIER: US 4355021 A  
TITLE: Virucidal wipe and method

**Abstract Paragraph Left (1):**

A substantially dry, impregnated wipe having therein iodine and means for retaining the iodine from escape therefrom, the iodine being present in an amount from about 1% to about 15% by weight of the wipe and in an amount sufficient to provide virucidal activity. The iodine is preferably present in an amount of from about 2% to about 5%. A flexible paper substrate is preferred, and the most preferred substrate is facial tissue. The iodine retaining means is preferably a suitable stabilizing surfactant present in an amount sufficient to complex the iodine, but preferably not substantially in excess of an amount sufficient to complex the iodine. A highly preferred stabilizing surfactant is polyoxyethylene (40) sorbitol septaoleate, present in weight ratio to iodine of from about 1:3 to about 2:1, and most preferably about 1:2.

**Abstract Paragraph Left (2):**

A method of reducing transmission of respiratory viral infections, which includes providing a substantially dry nasal wipe material of the type described and contacting such material with nasal surfaces in the presence of nasal discharge, and, preferably, thereafter wiping to dry hand surfaces with such material.

**Brief Summary Paragraph Right (28):**

Iodine is present in the wipes of this invention in an amount of from about 1.0% to about 15% by weight of the impregnated wipe. The iodine is most preferably present in an amount of from about 2% to about 5% by weight of the impregnated wipe. If the iodine level is below about 1% by weight of the impregnated wipe, the virucidal capabilities of the wipe may be insufficient. Iodine levels in excess of about 15% could tend to stain the skin, cause an irritating pungent odor, or weaken the paper substrate. When iodine is present in an amount within the preferred range of 2-5%, the most effective balance of efficacy, stability, cosmetic concerns, and sensory concerns is achieved.

**Brief Summary Paragraph Right (30):**

Several surfactants have been found to perform satisfactorily as the stabilizing surfactant in this invention. These include: linoleic diethanolamide, a nonionic surfactant available from Mona Industries, Inc., Patterson, N.J., under the trademark MONAMID 15-70W; the sodium salt of undecylenic acid monoethanolamide sulfosuccinate, a nonionic surfactant available from Rewo Chemical, Inc., Mauldin, S.C., under the trademark REWOCID DU-185; an ethoxylated polymethylsiloxane having nonionic surfactant qualities available from Union Carbide Corporation, New York, N.Y., under the trademark L-77 Silicone; and ditallow (diethylenetriamine) ethyleneoxide dimethyl sulfate quaternary, a cationic surfactant available from Sherex Chemical Company, of Dublin, Ohio, under the trademark VARISOFT 222. A highly preferred stabilizing surfactant is polyoxyethylene (40) sorbitol septaoleate, a nonionic surfactant available from ICI Americas, Inc., Wilmington, Del., under the trademark ARLATONE T.

**Brief Summary Paragraph Right (39):**

Ethyl alcohol is a preferred fugitive solvent for use in the production of the virucidal wipe of this invention. Ethyl alcohol dissolves the iodine crystals and stabilizing surfactant easily and vaporizes quickly during the drying step. Other fugitive solvents could be used, including alcohols other than ethanol, diethyl ether, water, glycerol, and mixtures of alcohol and water. Acceptable fugitive solvents must adequately dissolve the iodine crystals and other constituents of the iodine intermediate, must be capable of drying in a reasonable period of time, and must be toxicologically acceptable.

Detailed Description Paragraph Left (1):

About 90% of this solution is used to impregnate 5500 feet of a three-ply creped wadding facial tissue of 12-inch width, using a printing process. This web is thereafter air-dried and converted into acceptable lengths to provide virucidal wipes. The resulting wipe has an iodine content of about 3% by weight and a stabilizing surfactant content of about 1.5%. The dry wipe has approximately the same softness and surface characteristics as a facial tissue of the prior art, and exhibits good virucidal activity to interrupt the spread of respiratory viral infections.

Detailed Description Paragraph Table (1):

\_\_\_\_\_ Resublimed iodine crystals 727.2 g ARLATONE T  
(polyoxyethylene (40) 363.6 g sorbitol septaoleate) Perfume 72.7 g Ethyl alcohol (absolute) 17.0 kg.

Detailed Description Paragraph Table (3):

\_\_\_\_\_ Resublimed iodine crystals 0.4 g ARLATONE T 1.2 g Perfume  
0.02 g Ethyl alcohol 98.38 g. \_\_\_\_\_

Detailed Description Paragraph Table (4):

\_\_\_\_\_ Resublimed iodine crystals 4.0 g ARLATONE T 2.0 g Perfume  
0.4 g Ethyl alcohol 93.6 g. \_\_\_\_\_

Detailed Description Paragraph Table (6):

\_\_\_\_\_ Resublimed iodine crystals 0.6 g REWOCID DU-185 (the  
sodium salt of unde- 0.6 g cylenic acid monoethanolamide) Perfume 0.06 g Ethyl alcohol 98.74 g.

Detailed Description Paragraph Table (7):

\_\_\_\_\_ Resublimed iodine crystals 0.8 g L-77 Silicone (an ethoxylated  
polymethyl- 0.8 g siloxane from Union Carbide Corporation) Perfume 0.08 g Ethyl alcohol 98.32 g.

Detailed Description Paragraph Table (8):

\_\_\_\_\_ Resublimed iodine crystals 0.6 g VARISOFT 222 (ditallow  
(diethylene- 0.6 g triamine) ethoxyleneoxide dimethyl sulfate quaternary) Perfume 0.06 g Ethyl alcohol 98.74 g.

Detailed Description Paragraph Table (9):

\_\_\_\_\_ Resublimed iodine crystals 1.1 g ARLATONE T 0.55 g  
Perfume 0.1 g Ethyl alcohol 98.25 g. \_\_\_\_\_

## CLAIMS:

1. In a method for removal of respiratory viral infection-related nasal secretions of the type including the step of wiping nasal surfaces with a dry, flexible, absorbent wipe material, the improvement comprising:

providing such wipe material having substrate means with impregnant therein which in contact with moisture will release iodine on said substrate means in an amount sufficient to provide virucidal activity, said wipe material being substantially free of moisture before use and said impregnant retaining the iodine from escape from said wipe material before use;

contacting said wipe material with nasal surfaces to remove nasal discharge therefrom, thereby moistening said wipe material to release on said wipe material a virucidally sufficient amount of iodine; and

thereby killing with said iodine viruses on the contacted surfaces,

whereby transmission of respiratory viral infection is interrupted without significantly modifying behavioral patterns of infected persons.

2. The method of claim 1 wherein said iodine on said dry wipe material is present in an amount of from about 1.0 to about 15% by weight of said dry wipe material.

4. The method of claim 1 including the subsequent step of wiping to dry hand surfaces with said wipe material.
6. The method of claim 1 wherein said impregnant includes iodine in an amount of from about 1.0 to about 15% by weight of said dry wipe and a suitable stabilizing surfactant in an amount sufficient to complex said iodine.
12. The method of claim 11 including the subsequent step of wiping to dry hand surfaces with said wipe material.